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The Modern

Cotton Shirting Fabric

An Account of Its Processing Methods

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IN SPITE of numerous efforts to convince men folk of the reputed advantages of knitted rayon shirts for all-round wear it appears that their use is at present mainly limited to sports and special wear where great elasticity is necessary. Most shirts are made from a plain weave fabric having a slight poplin character and each year some millions of yards of this type of fabric are used for shirt making. To the bleacher, mercerizer, and finisher this fabric is therefore an item of considerable importance.

The specification of the shirting fabric here considered is simple. As stated previously it is plain woven. The warp threads are fairly closely packed—about 120 to 130 per inch—and they are twisted moderately hard. The filling threads are soft twisted and of lower counts than the warp, but they are less tightly packed—about 70 to 80 per inch. Often the weft threads are white and the warp threads colored so as to produce warp stripes. Sometimes both warp and weft threads are colored so as to give a plain colored or even a marl effect. It is very seldom that the fabric has filling stripes only. The finished fabric must have a silky handle and a high luster. As far as possible it must resemble real silk fabric. On this account the fabric is nearly always mercerized in the piece.

Most shirting fabric is given a specially shrunk finish so that the shirts and soft collars made from it can be guaranteed unshrinkable. But in order to avoid the extra costs of this method of finishing it is often sufficient to finish the fabric under ordinary conditions except that stretching is avoided as much as possible. In this manner a fabric is obtained which shrinks only slightly on repeated washing and usually gives nearly equal satisfaction to the specially shrunk fabric.

At the present time the bleaching and finishing prices for this type of shirting fabric are very keenly cut, so that while nothing but the best finish will be tolerated, all unnecessary costs must be rigidly eliminated. This fact intimately affects the processes adopted.

It is interesting to note that a considerable amount of the highest quality fabrics of this type are manufactured and finished in Great Britain and exported to America in spite of import duties. There must be some fundamental reason for this. Is it that Great Britain is able to produce a superior article not equalled in America? Certainly some of the fabrics imported into America have a super silky handle, and to assist this property the fabric is made with the most expensive Sea Island cottons. They are more costly than the higher luster rayon fabrics.

In most of these shirting fabrics there are white portions so this makes it necessary to give the fabric a thorough bleach in order that such parts may have a good white color. Sometimes it is only the selvedge which is white and for this comparatively useless portion of the fabric it is necessary to carry out a full chemick treatment. This seems ridiculous but nevertheless is a fact. But since bleaching is generally necessary, the colors woven into the fabric must obviously be fast to bleaching. Usually the fastest vat dyes are used for coloring the yarns before weaving, but in order to avoid expense in yarn dyeing the somewhat cheaper naphthol dyes are sometimes employed. Difficulties sometime arise in the bleaching and finishing works on account of the use of loose and therefore unsuitable dyed yarns in the fabric. So there must be careful watch on the fastness of the fabric or disputes may easily be occasioned.

Having the above facts in mind it is now possible to turn to the actual processing methods which have proved capable of giving excellent and satisfactory results.

Briefly the processing divides itself naturally into three stages:—

1. *Bleaching*: singeing, scouring, chemicking.
2. *Mercerizing*: mercerizing, scouring and soaping.
3. *Finishing*: drying, stentering and calendering.

1. BLEACHING

In the bleaching process it is essential to remove the greater part of starchy and nitrogeneous impurities so

that the caustic alkali used in mercerizing may be most efficient in producing a high luster. Also the fabric must be made a good white in those parts free from colored yarns. These results would, with a plain fabric, be accomplished by giving it two good kier boils followed by a treatment with chemick liquor. It is not possible to apply these processes to colored shirting fabric since, in kiering, the colored stripes would most probably mark off on to the white parts. It must be remembered that vat dyes are rendered slightly soluble when heated with cotton and caustic alkali (these conditions obtain in kiering) owing to the fact that an alkali and cellulose together act as a reducing mixture. Hence the desirability of avoiding kiering for this type of fabric. The starch and nitrogeneous impurities must therefore be removed by a simpler process of scouring in which the fabric does not lie wet and alkaline in a compressed folded condition such as would promote marking-off. Scouring will therefore be carried out in rope washing machines.

The bleaching processes will comprise singeing on one or both sides of the fabric, followed by impregnation with a starch-liquefying enzyme, scouring with dilute soda ash solution in rope washing machines, chemicking with bleaching powder or sodium hypochlorite solution, washing, souring, and a final thorough washing with water. It is seldom necessary to use an antichlor after chemicking.

Singeing may be effected with hot copper plates but it is quite sufficient to use the ordinary gas flame singeing machine. After leaving the flames the fabric is directly led through a padding machine containing the starch-liquefying enzyme solution and piled down in heaps or plaited down in open width. In this manner the padding liquor serves the dual purpose of making soluble the impurities in the fabric and also of quenching any sparks from the singeing flames which may chance to fall on the traveling fabric.

The fabric, saturated with starch-liquefying liquor, should not be piled into large heaps since, during the reactions which take place, a considerable amount of heat is developed and this would lead to reduction of the vat dyes in the colored stripes with consequent marking off. It is therefore necessary to pile or plait the fabric so that there is a good circulation of air through it; this promotes cooling of the fabric. The concentration of starch-liquefying enzyme in the padding liquor should be high enough to allow complete solubilization of the starch impurities within two hours.

Afterwards, the fabric is led in rope form through two or three (according to their size) rope washing machines containing detergent liquors at the boil. The first should contain boiling water, the second a boiling solution of soda ash, and the third cold water. At this stage the color of the fabric may be somewhat browner than in the grey state but in spite of this it contains less impurities. The brownish color is easily removed with chemick.

Following the wash with water, the fabric should be led through a fairly hard nip so as to remove as much moisture as possible and then led through a padding mangle containing the chemicking liquor. It is then piled into cisterns and allowed to lie there until the whole of the fabric has had one impregnation. The fabric is then led again through the chemicking machine for a second impregnation with chemicking liquor. There is a definite reason for this double impregnation and in fact it is necessary although it adds to the cost.

If the fabric is led through the chemicking liquor but once and then piled, the bottom pieces of the pile are in contact with the liquor for a much longer period than the top pieces. It usually takes at least one hour to run one lot of fabric through the chemicking padding machine. But by padding twice, the whole of the fabric is subjected uniformly and equally to the action of the chemicking liquor, and the first pieces are bleached to the same extent as the last pieces.

Afterwards, the fabric is led through the rope washing machines provided with running cold water so as to remove completely all residual chlorine, and is then led over a scutcher and dried on the usual drying cylinders. The fabric is then ready for mercerizing. But before dealing with this second stage in the processing it will be necessary to make mention of one or two important factors in the bleaching process.

It must be remembered that most fabric of this shirting type contains colored stripes and other effects and therefore it is most essential that these colors be not diminished in intensity or changed in tone. A chlorine bleach can, of course, be very destructive towards even vat and naphthol dyes. If the process is not controlled carefully it will be found that on some days the colors are affected while on other days the colors will appear to resist the bleaching very well. Also, it is a matter of experience that the stripes occasionally appear to cause tendering of the fabric when this would not, from a consideration of the concentration of chlorine used, be expected.

Firstly, attention will be given to the preservation of the colored stripes. It is found that the fastness of vat dyed cotton towards chemicking is much influenced by the pH of the bleach liquor. If cottons dyed with different vat dyes be subjected to bleaching with chemick liquors of pH varying from say 5 to 13 and then compared, it will be found that generally the best results are obtained with liquors of pH = 8 to 11. At the same time it requires a longer period of bleaching to obtain white fabric at pH= 11 than at pH= 8, and this is a factor which must be considered. From experience with a large amount of this shirting fabric it is concluded that bleaching with a liquor about pH = 8.5 to 9.0 gives the best results. Also bleaching with an acid or neutral chemick liquor can easily give marked changes in the colored stripes.

The bleaching liquor should therefore be not only started at pH = 8.5 to 9.0 but it should be maintained at this degree of alkalinity during the whole of the bleaching process. The effect of bleaching is to produce acid substances (by oxidation of impurities in the cotton fabric

to organic acids) in the chemicking liquor and so gradually reduce the pH. It is therefore necessary to add to the chemicking liquor from time to time a small amount of an alkali, preferably sodium carbonate (if sodium hypochlorite solution is used). In those cases where a solution of bleaching powder is employed it is probably better to add milk of lime to increase the pH of the chemicking liquor.

As a means for determining the pH of the chemicking liquor the B.D.H. Universal Indicator has been found quite satisfactory. The test is conducted in the following manner. Firstly a neutral 1% solution of hydrogen peroxide is made by adding a few drops of caustic soda to the commercial solution which usually contains an acid for stabilizing purposes; phenolphthalein is a useful indicator for this purpose. Then two or three c.c. of this neutral hydrogen peroxide solution is added to five c.c. of the chemicking liquor under test and the mixture heated until all the liberated oxygen is evolved. At this point the active chlorine in the liquor has been destroyed according to the following equation:

$$H_2O_2 + Cl_2 = 2HCl + O_2$$

The liquor can then be cooled and to it added one or two drops of the Universal Indicator. The pH can then be noted from the color developed in the liquor.

It may be noted that the destruction of the active chlorine is necessary since it would otherwise attack the Universal Indicator and interfere with its color reactions.

The strength of the chemicking liquor will to some extent be dependent on the rapidity with which bleaching is required to be effected. It has been found satisfactory if the chemicking liquor contains 0.1 to 0.4 grams of active chlorine per liter.

As regards tendering of the fabric, two causes have been discovered. Certain yellow to red vat dyes have the property of accelerating the action of the chemicking liquor in daylight and particularly strong sunlight. Also such acceleration can be promoted by sunlight itself in the absence of dyes.

Derrett-Smith and Nodder (J. Text. Inst., 1932, *23*, 293 T) have examined this harmful influence of vat dyes very carefully and their results are worthy of the attention of all bleachers of colored cotton goods. Among the dyes which promote tendering are Cibanone Orange R, Cibanone Yellow R, Caledon Gold Orange GS, and Indanthrene Rubine R, and it is also remarkable that this catalytic action is most marked when the chemicking liquor is about neutral. The minimum effect is observed when the chemicking liquor is not less than pH = 9.5.

Thus in bleaching cotton shirting fabric it is advisable to ensure that the fabric is not exposed to strong sunlight. When tendering does occur around the stripes then examination of the dyes present should be made in order to ascertain whether one or more of the unsuitable dyes have been used by the yarn dyers. Today, most yarn dyers know which dyes are unsuitable for use with cotton yarns to be used in shirting fabrics and so they avoid the use of these.

But sunlight accelerates the action of a chemicking liquor even when no dyes are present, and it is the experience of practical bleachers that cotton goods can be tendered by allowing the fabric, impregnated with the chemicking liquor, to lie exposed to sunlight coming directly from an overhead window. It is therefore desirable for the bleach house illumination to be of moderate intensity and to be free from shafts of strong sunlight.

Many shirting fabrics contain black stripes and difficulties occur when the cotton in these stripes is dyed with Aniline Black produced by the copper process; the yarn then contains small traces of copper. It is frequently found that such black stripes become very tender after chemicking so that the warp threads readily break. This is due to the catalytic influence of the copper since this causes the chemicking liquor to attack the cotton very rapidly.

It is not wise to use Aniline Black dyed cotton for shirting fabric which has to be well bleached (that is, a fabric which has a large proportion of white) on account of this tendering. The stripes should be produced with yarn dyed with a naphthol or vat black, although care has then to be taken (especially with the naphthol black) that bleeding does not take place while the fabric is saturated with alkali. Often the vat black dyed yarn is greenish in color in the grey fabric but the chemicking rapidly oxidizes this to a deep black.

The production of deep brown and red shades on cotton yarn with vat colors is somewhat expensive and for this reason yarn dyers frequently use the naphthol dyes as being cheaper. The red shades are usually found to be fast to chemicking but care must be taken with the brown shades as these occasionally lose depth of shade.

Grey stripes obtained with vat dyed yarns are also liable to turn thinner and more violet on chemicking. On the whole it may be stated that this color is one of the least satisfactory.

Considering the bleaching of colored cotton shirting fabrics from a broad viewpoint it is surprising how many millions of yards can be processed, using reasonable care. without serious difficulty. It is only occasionally that trouble is experienced and this can always be traced to the use of an unsatisfactorily dyed yarn or a lapse in the control of the chemicking liquor. Generally it is not possible to test the fastness of color of all the grey fabrics before processing and it must be assumed that the manufacturer is using the usual and suitable colors. But it is a wise precaution to keep small cuttings from the various consignments so that if difficulty occurs through bleeding or discharge of the colored stripes the original fabric can be examined and so allow the blame to be placed on the right shoulders. Of course, when new fabrics are introduced, they should be first thoroughly tested in the laboratory. Also it is recommended to sew about one-half yard of the new fabric on to other fabric passing through the works

processes and so ascertain whether its fastness is satisfactory.

One other point in connection with change of color in the stripes should be mentioned. It must always be remembered that mercerization follows bleaching and that this process also affects the colored stripes. Generally mercerization causes the colors to appear deeper, this probably being due to the swelling of the cotton yarns which mercerization produces. So that if, after bleaching, some of the colors appear somewhat thin there is always a possibility that these colors may be restored in the mercerizing process.

Most blue vat colors are turned slightly greenish by chemicking but mercerization assists the restoration of the true color. Also, as will be indicated later, there is a method for restoring blue stripes to their redder appearance if the greening has not been carried too far.

2. MERCERIZING

It has been noted that the fully bleached fabric is dried before mercerizing. This has been found desirable from the viewpoint of securing the utmost increase of luster in the fabric. Some mercerizers might feel that the cost of drying could be eliminated and the bleached fabric merely mangled and then mercerized in the wet condition. The writer has found however from experience that the best results are obtained from the dried fabric.

For mercerization it is possible to use either of two types of machine—the stenter type provided with clip chains and the chainless type having numerous expanding rollers. Possibly both types are satisfactory but certainly excellent results can be produced by means of the stenter type.

A mercerizing machine of the stenter type should be provided with two padding mangles with a number of freely rotating steel drums between. Then follows the stenter portion of the machine where the alkali-impregnated fabric is stretched to width while being washed from alkali by means of three or four overhead spurt pipes with accompanying suction boxes underneath the fabric. Finally the fabric is led through a box provided with water acidified with sulfuric acid so that all residual alkali is neutralized, and then through two more washing boxes for the removal of all acid. The fabric is then ready for drying and finishing.

Mercerizing forms an important stage in the treatment of a shirting fabric. It is essential to secure the highest increase of luster combined with softness of handle. At the same time nothing must be done to the fabric to make it more shrinkable when finished.

Attention will be given to luster increase first. Before mercerizing, the fabric has been thoroughly freed from impurities by bleaching so that it will have good absorbent power towards the caustic soda solution used. Also since starch substances are not coating the warp threads they will not swell in the mercerizing liquor and so prevent full mercerization of the cotton fibers. With the aid of two impregnating mangles it is also possible to force the caustic soda liquor right into the interstices of the fabric and so ensure perfect penetration to all the fibers.

The maximum luster increase in cotton fabric during mercerization is obtained by powerful stretching while the fabric is impregnated with alkali. This stretching can be secured very conveniently by making use of the two mangles as shown in Fig. 1. Each mangle A and B contains three bowls working under hydraulic pressure; the middle bowl is of hard rubber or vulcanite and the two outer bowls are of steel. The fabric F passes through the first mangle A. then around the cylinders C, through the second mangle B and then on to the stenter frame to be stretched in width and washed free from alkali. The speeds of mangles A and B can be varied independently of each other.

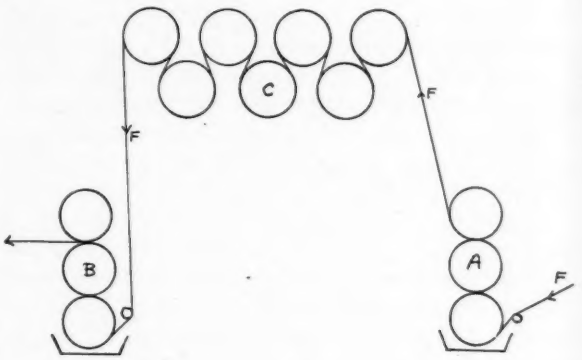

Fig. 1

By means of the arrangement of mangles and cylinders described it is possible to produce a very powerful warp stretching on the fabric. While the fabric is passing from mangle to mangle the second mangle B can be speeded up slightly so that it runs faster than A. This causes the fabric to be stretched and the amount of stretching is governed by the increased speed of mangle B. In practice the stretching should be carried almost up to the breaking point of the fabric so that this appears as "tight as a drum" while passing between the mangles.

As might be expected it is somewhat more difficult to impregnate the fabric with caustic coda while under such a high warp tension. For this reason it is advisable to have present in the mangle troughs suction devices for drawing the alkali through the fabric. In a device of this type which has proved quite satisfactory the trough is provided with a horizontal pipe (about 1½ inches diameter) extending the whole length of the mangle trough and submerged in the caustic soda solution. This pipe has on its upper side a slot also extending its whole length. The pipe is connected to a rotary pump. As the fabric is led through the trough it passes over the slot while the caustic coda is continuously drawn through it into the pipe and thence through the pump for return to the trough. By use of this suction device excellent penetration of the fabric by the mercerizing liquor is secured.

(Continued on page 298)

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American Association of Textile Chemists and Colorists

MAY 20, 1935

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CALENDAR OF COMING EVENTS

Meeting, New York Section, Chemists Club, New York City, May 24, 1935

* * *

Outing, Philadelphia Section, Torresdale Country Club, Torresdale, Philadelphia, June 7, 1935

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Analytical Methods for a Textile Laboratory

The Council of the A.A.T.C.C., believing that Dr. Scott's series of articles entitled "Analytical Methods for a Textile Laboratory" might well be brought together in book form, have proceeded with its publication. Copies may be obtained from the Secretary of the Association, Harold C. Chapin, Lowell Textile Institute, Lowell, Mass., for 75 cents.

Proceedings of the American Association of Textile Chemists and Colorists

Freezing Process

for Cleaning Wools of Foreign Matter*

By R. J. WIG†

ORIGIN

ABOUT three and one-half years ago a wool buyer, who was familiar with California wools, met a mechanic in Los Angeles who had been employed to develop a new shoddy machine. The wool buyer remarked to the mechanic that if he had any inventive ideas he could employ his time to better advantage than trying to develop a new shoddy machine, by developing some machine that would mechanically and effectively remove mestiza burrs from wool. He explained to the mechanic that the mestiza burr grew extensively in California and other western states, also in Australia, South America and South Africa and was found in many wools from these districts; the burr was from one-eighth to one-quarter inch in diameter, constructed like a coiled watch spring with thorns along the coiled strand; the seeds inside the burr make excellent feed for sheep.

In the grease wool these mestiza burrs are matted into the fleece and somewhat entangled. If the fleece wool is scoured, the grease, suint and dirt are removed from the fiber but the burr remains and becomes even more entangled with the wool fibers.

Several weeks after the wool buyer met the mechanic and made this suggestion, the mechanic was on a picnic with his family. They had some ice cream packed in dry ice. A blanket had been spread on the ground and the dry ice, when removed from the ice cream, was thrown onto the blanket. In picking up the blanket after their lunch, the mechanic observed the vegetable matter on the ground was frozen to the blanket and when he shook the blanket the vegetable matter fell away, like ice particles. This incident suggested to him the possibility of freezing the burrs in the wool and then removing them by some shaking device, burr picker or duster.

After a little experimenting, the mechanic conceived the idea of wetting the wool or scouring the fleece wool to remove the grease and of immediately freezing it as the wool came from the squeeze rolls of the scouring bowl, with the burrs and vegetable matter more or less saturated with water. The moisture in the form of small globules left on or between the wool fibers would freeze into minute ice particles and the burrs and vegetable matter being water absorbant would freeze into solid particles. These solid particles, in reality small heavy ice particles, would then by their weight be thrown out from the wool in a duster or similar machine.

The inventor's original idea contemplated making this process applicable to defective clothing wools only, which might normally be carbonized, and therefore it would be permissible to pass this frozen wool through a burr picking machine.

PRELIMINARY EXPERIMENTS

The inventor conveyed his idea to the wool buyer and together they secured the cooperation of a manufacturer of wool felt, who had a burr picker, and several tests were made by packing the machine in dry ice and freezing the wool with dry ice. The results of these tests were sufficiently promising to encourage the enlistment of financial aid from a local firm of engineers, who financed the renting of space in a cold storage plant and the renting of equipment to carry on more comprehensive tests. This firm spent several thousand dollars and a number of month's time during which they processed small quantities of different wools, enough to determine that the process had possibilities, but it was apparent a large amount of time and money would be required to perfect the process.

At this stage the project was brought to us and after investigation we undertook its further development. Space in a cold storage plant was rented at Long Beach, California, engineers and experienced wool men were engaged, machinery was purchased and after several months a few hundred pounds of a number of different wools were treated. The process appeared to be successful in removing burrs and vegetable matter, but we did not know how much damage, if any, had been done to the wool fiber, nor whether the cleaning which had been done was sufficiently thorough to be worth the expense.

Samples of these treated wools were brought East and shown to the executives of several wool companies and the technical staff of some of the textile schools. We were greatly encouraged and told if we could duplicate on a commercial scale the samples we exhibited, the process had merit.

*Paper presented at Meeting of N. N. E. Section, March 8, 1935.

†Vice-President, Frosted Wool Process Company.

DEVELOPMENT OF PROCESS

After several conferences, arrangements were made with the Lowell Textile Institute, Lowell, Mass., to continue the development work and perfect it on a commercial scale at this institution, where we would have available all the necessary testing facilities, as well as the advice and council of the technical staff of the institute and cooperation of the various wool companies but a few miles distant from Lowell.

We wish to express our appreciation to the executives of the Lowell Textile Institute and especially to Professor E. H. Barker, R. L. Brown, James H. Kennedy, Jr., and John C. Lowe of the wool department, and the entire staff of the Institute for the very friendly, helpful, encouraging cooperation they have given us, that has made possible the further perfection of this process.

Professor Barker suggested when we first started our work at the Institute in June, 1932, that we direct our efforts toward perfecting the process for use on worsted wools, in that this was the large consuming market for virgin wools.

We purchased a five-ton York refrigerating unit that was capable of giving temperatures as low as 10 degrees below zero F. and freezing two hundred pounds of wool per hour. A refrigerating chamber was constructed and auxiliary equipment designed and built.

The process as developed at the Long Beach, California, experimental plant consisted of sorting the wool, scouring it, wringing out the surplus water, and while in a moist condition placing the wool on a freezing belt through which was blown a blast of cold air and, after freezing, feeding the wool containing the frozen vegetable matter into a burr picker or duster, or a combination of both, where the vegetable matter and dirt would be shaken out.

The first plant erected at the Lowell Textile Institute in June, 1932, was constructed to operate in a similar manner by wetting the scoured wool, freezing it and shaking out the foreign matter in a dusting machine.

The wools containing mestiza burrs with which we had worked in California were apparently a Fall clip of wool. The burrs were large, hard and mature. The wools with which we started to work in the Lowell Textile Institute were apparently Spring clip and contained many small soft green mestiza burrs. We were disappointed to find that these soft burrs would not absorb water and when frozen remained soft and flabby, so they could not be dislodged from the wool upon shaking in the dusting machine. Various methods were tried to wet these burrs; chemical treatment to obtain water penetration was resorted to, and while an improvement was obtained, the results were not entirely satisfactory.

While this work was under way, the suggestion was made that we try freezing the grease wool before scouring and without wetting the wool. This was tried and to our surprise the burrs and vegetable matter were removed substantially as effectively as though the wool had been previously wet.

After some investigation it was determined that the burrs were less badly tangled in the wool fibers in the fleece wool than after the wool was scoured. Also there was sufficient natural grease from the sheep, coated on the vegetable matter and wool fibers, so that, if this grease is frozen to a hard brittle condition and subjected to impact as it is in a duster, the grease shatters into fine dry powder, releases the hold of the fiber on the burr or vegetable matter, or foreign matter, and in this condition the foreign matter including the burrs may be readily separated from the wool in the duster.

The lowest temperature we could reach with our single stage York compressor was about 10 degrees below zero F. and this did not appear to be low enough to make grease entirely dry and brittle, so we installed a York Booster which made it possible to carry the temperature in the refrigerator to 30 degrees below zero F. With this lower temperature we were able to substantially remove the vegetable matter from every wool with which we worked. I say substantially because we do not completely remove all the vegetable matter, but what is not completely removed is usually detached from the fiber so that most of it readily falls away when the wool is processed in carding, or other subsequent operations. With most wools if desired the vegetable matter can be practically wholly removed from the wool but only with some sacrifice of fiber length and loss of fiber.

ALL FLEECE WOOLS CONTAIN VEGETABLE MATTER

About this time it was recognized, after conference with a number of the wool companies, that the mestiza burr was not the only troublesome form of vegetable matter in wool. It may be interesting to you to see some of the more common forms of vegetable matter found in wool. (Figures 1 to 15.)

All wools contain more or less vegetable matter. Contrary to the common opinion of some, the vegetable matter is not completely removed by the present mechanical processing equipment of the mill. Even the so-called free wools contain from one half per cent to three per cent of vegetable matter on the weight of fleece wool and much of this is found to be more or less troublesome in increasing processing costs and causing defects in the finished fabric.

It was pointed out that it would be of much value to all handlers of wool if the foreign substances could largely be removed from wool immediately after sorting and before the manufacturing processes were started. Following this suggestion and with the encouragement of some of the wool manufacturers, efforts were directed toward

making the process applicable to all combing and clothing wools including the so-called free wools.

DEVELOPMENT OF EQUIPMENT

With this equipment we could only process one to two hundred pounds per hour which was not commercial, so our next job was to develop equipment that would process a minimum of 1,000 pounds per hour.

The better part of a year was spent experimenting with different cleaning machines to develop one that would have a capacity of 1,000 pounds per hour and efficiently clean without materially damaging the fiber or knotting or rolling the wool.

The machine we are now using is the eighth one we have constructed. This research involved testing standard types of dusting machines, determining their faults, working out a design that would not roll or knot the wool, determining the effect of different sizes and shapes of pins, their proper spacing, the effect of dead pins, character of screens, proper clearances, effect of speed of rotation and many other factors upon which we spent the better part of a year.

In connection with this research we found a stroboscope most helpful. With this instrument it is possible to optically stop and study any of the moving parts while in motion. This is done by synchronizing the flash of a mercury vapor lamp with the period of rotation or motion of the part of the machine being observed by which, therefore, you always see the object at the same location on each rotation and the object has the appearance of standing motionless. With this instrument one can actually see the movement of the wool on the pins and in the duster while in motion.

An additional set of problems were also involved in designing a machine to operate at low temperatures. After extensive research, a satisfactory cleaning machine was developed.

Our present cleaning machine has a capacity of 1,000 to 1,500 pounds per hour on the normal grades of quarter blood to fine combing and clothing wools. With some extremely defective wools this capacity may be reduced. It will not roll nor knot the wool. In the low temperatures the screens remain clean indefinitely. The machine is so constructed that the performance is sensitive to adjustment which gives the operator great leeway in the treatment which he may give the wool.

Before proceeding further with our development work, tests were made on combing wools at the Lowell Textile Institute to determine the effect of the frosted wool process on the production of noils. The results of some of these tests are shown in Table No. 1, Figure 16, from which it may be seen that the percentage of noils is practically identical for these particular wools and frosted treatment. Likewise tests were made to determine the breaking strength of yarns. The results are shown in Table 2,

KEY TO ILLUSTRATIONS

Appearing on page 273

Fig. 1—Common cockle-burr (*Xanthium canadense*). Has no feed value and is one of the most universal pests to the wool grower. These burrs are easily removed by processing machinery.

Fig. 2—Spiny cockle-burr (*Xanthium spinosum*). Has a low feed value and is found commonly in Central California. It is easily removed by processing machinery.

Fig. 3—Grease wool with entangled cockle-burrs.

Fig. 4—Foxtail or Squirreltail grass (*Hordeum murinum*). Is an excellent feed up to time of seed formation. It is one of the most common and most troublesome forms of vegetable matter and very difficult to remove by normal wool processing equipment.

Fig. 5—Pip-gut or broncho grass (*Bromus rigidus*). Is found widely distributed and is very difficult to remove from wool, the fiber-like strands carrying through all the processing machinery with the wool fibers.

Fig. 6—Grease wool with entangled grass fibers.

Fig. 7—Grease wool with entangled grasses and straws.

Fig. 8—Blue-stem Filaree (*Erodium Botrys*). Has excellent feed value but the corkscrew filament is very troublesome in the wool.

Fig. 9—Red-stem Filaree (*Erodium Cicutarium*). Has excellent feed value but the corkscrew filament is very troublesome in wool.

Fig. 10—Grease wool with entangled Filaree or corkscrew burrs.

Fig. 11—Burr clover or Mestiza or spiral burr (*Medicago hispida*). Is probably the most troublesome and difficult to remove of all forms of vegetable matter. It is found in certain districts of many wool producing countries. The burr uncoils as the wool is processed and the strands which are barbed often carry through into the finished yarn. The seeds in the burr have excellent feed value.

Fig. 12—Grease wool with entangled clover or spiral burrs.

Fig. 13—Yellow star thistle (*Centaurea Solstitialis*). Is a soft burr that pulls apart and distributes in the wool and is troublesome to remove.

Fig. 14—Grease wool containing soft thistle burrs.

Fig. 15—Hoarhound (*Marrubium vulgare*), the ripened seeds of which are often found in the wool fleece. It is not particularly troublesome for the seed readily drops out in normal wool processing.

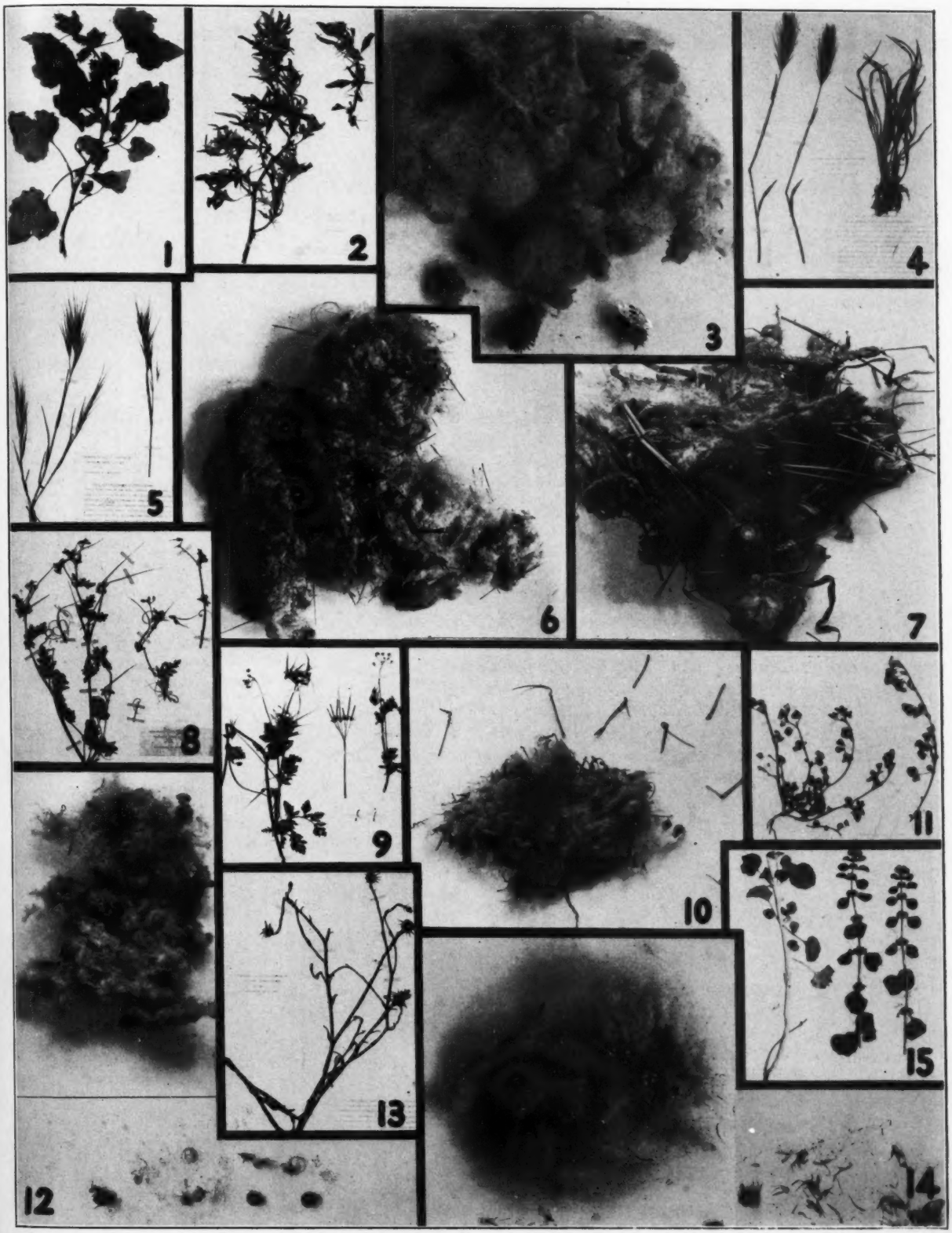
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TABLE NO. 1

Percentage of Noils from Combing Tests of Several Typical Wools Untreated and Treated by Frosted Wool Process

Sample No.	Description	% of Noils on Top Plus Noils	
		Untreated	*Treated*
200	64's matchings	8.96	8.61
600-15	Fine to high ½s	12.84	12.64
220	High ⅜s matchings	5.02	4.94
530	⅜ to low ½s	14.24	14.79
620	⅜s domestic	14.04	14.15
601-84	⅜ to low ¼s	10.78	10.63

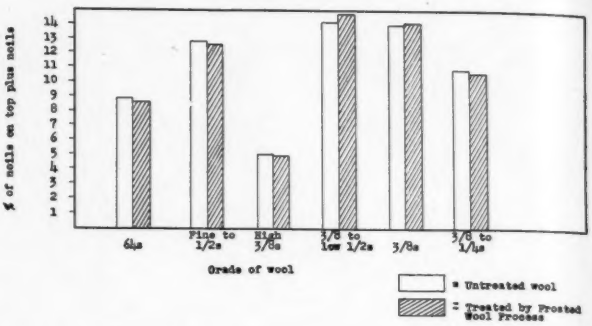


Fig. 16—Percentage of Noils from Combing Tests of several Typical Wools Untreated and Treated by Frosted Wool Process

TABLE NO. 2

Breaking Strength of Yarns Made from Wools Untreated and Treated by Frosted Wool Process

(10 *skeins*—80 *yards,* 80 *loops conditioned to* 65% *RH*)

Sample No.	Description	Break in Pounds	
		Untreated	*Treated*
200	64s matchings Spun 1/16s	84.2	67.6
200	64s matchings Spun 2/16s	201.4	182.5
220	⅜ matchings Spun 1/16s	57.9	60.4
220	⅜ matchings Spun 2/16s	175.3	174.9

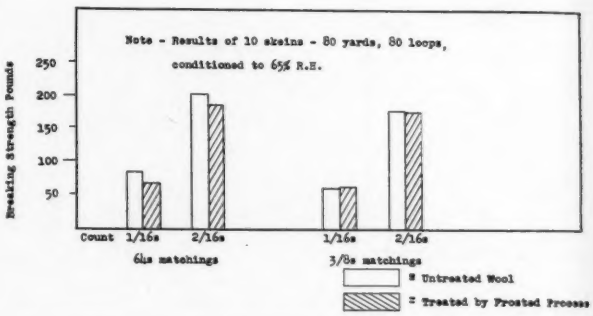


Fig. 17—Breaking Strength of Yarns made from Wools Untreated and Treated by the Frosted Wool Process

Figure 17. While some difference in strength was shown, it was not great.

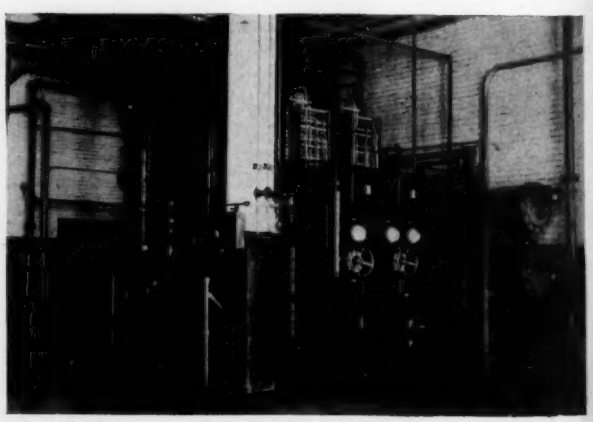
Dyeing tests were also made at the Lowell Textile Institute with a large number of dyes and no material difference was observed in the frosted and untreated wool. Some of the dye tests in the mills, however, have shown deeper, brighter and clearer colors with some wools after the frosted treatment.

In order to determine if the individual fibers were changed physically by the frosted wool process, Professor Edward R. Schwarz of Massachusetts Institute of Technology, who is well known as a microscopist on textile fibers, was engaged to make an examination of wool fibers that had been subjected to the freezing process and similar fibers that had not been exposed to the freezing process. His conclusion was that there was no apparent physical change in the fiber nor in its structure.

After perfecting the cleaning machine, we needed more freezing capacity and this called for an entirely new plant. Upon the advice of several mill operators, we decided to place the commercial size plant in the Lowell Textile Institute to completely work out all the problems of operation before installation in a mill or commercial operating plant. We were pleased this decision was made for we found several features of the commercial installation that needed further research.

The full size commercial frosted wool process unit consists of a Vilter Mfg. Co. compressor and rotary booster refrigerating unit with about a 27-ton rated capacity providing an air temperature of 40 degrees below zero F. It is capable of operating to temperatures as low as 60 degrees below zero F., but it will not continuously handle the full wool load at this minimum temperature. (Figure 18.) Any standard make of refrigerating equipment may be used.

Fig. 18—View of Refrigerating Equipment Used for Providing Low Temperature for the Wool Freezing Chamber



The cleaning plant consists of a refrigerating room (Figure 19) about 40 feet long by 12 feet wide and 12 feet high. Its shape can be modified to meet the conditions of the space available.

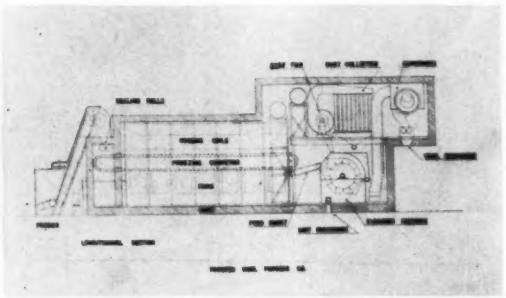


Fig. 19—Longitudinal Cross-Section of Wool Freezing Chamber showing Processing Equipment

The machinery consists of a standard wool feeder, 6 feet wide, which conveys and elevates the wool after sorting and without any preliminary opening to the top of one end of the refrigerated chamber. The wool drops into the cold chamber through a pair of sealing rolls, which excludes the free flow of warm air, into the freezing chamber.

From the sealing rolls the wool drops onto a six foot wide freezing conveyor, which in the present case is a modified Sargent dryer, 20 feet in length, the coils above the freezing belt carrying the refrigerent in place of steam. The blast of air is driven through the refrigerated coils and through the wool on the conveyor, the air being constantly recirculated as in a Sargent dryer.

The wool drops from the end of the freezing conveyor onto a short wood slat feeder apron, which carries the wool into the duster or cleaning machine. The feeding apron and discharge door of the cleaning machine are automatically controlled so that the charge of wool is retained in the cleaning machine for a previously determined number of revolutions. The actual cleaning is an intermittent batch operation, so it can be definitely controlled but the actuating device is automatic, so it handles the wool continuously as it comes from the freezing conveyor.

There is no operator in the freezing chamber during the processing of the wool. The machines are synchronized and automatic, with an automatic electrical control so if anything should interfere with the free flow of wool through the machine, the machine would automatically stop. It is normally operated at a temperature between 25 degrees and 35 degrees F. below zero.

The amount of treatment in the cleaning machine is determined by the character and quality of vegetable matter, by the character of the wool and by the results sought. All machine adjustments are made by the operator to handle the particular wool to be treated. The period of treatment, which materially affects the amount of cleaning, may be changed without interrupting processing. This control of the processing may be located in the superintendent's office or at any location desired where it will be accessible only to the authorized person.

The clean wool discharges from the cleaning machine onto a conveyor, which carries it to the suction duct of the wool condenser, from the condenser it is discharged out of the refrigerated chamber through sealing rolls. Since the wool is in the cleaning machine only a few seconds some particles of vegetable matter that were detached from the wool in the cleaning machine do not have time to get out of the chamber holding the clean wool, before the clean wool is discharged from the cleaning machine. Therefore some of these loose particles of vegetable matter discharge from the cleaning machine with the clean wool and to further remove these, subsequent conveyors are so devised as to perform the dual function of removing a large portion of these loose particles, as well as conveying the wool.

The wool is dry, but in a fluffy open condition, ideal for scouring, as it is discharged from the refrigerated chamber and, after passing over the screen, it may be immediately dropped directly into the scouring bowl, or transported to bins for storage, or packed in bags for shipment.

The dirt, vegetable matter and grease, all in a dry condition when removed from the wool, are collected through conveyors, and by a fan and a cloth bag dust collector, and discharged from the refrigerated chamber by means of a screw conveyor. These waste products may be disposed of by burning under the boilers or otherwise if desired.

The grease removed from the wool in a frozen state is dry and not sticky as at normal temperatures, which is an important factor in keeping the screens of the cleaning machine clean and making it possible to collect the grease dust in a cloth bag dust collector.

Figure 20 shows another arrangement of the equipment. The equipment is of such size that, if desired, it can with a slight rearrangement be installed in place of the first scouring bowl of a train. There are several units to the equipment which permits rearrangement to meet almost any reasonable space requirement. Photographs of the freezing chamber, and feeder of the installation at the Lowell Textile Institute are shown in Figures 21 and 22.

We are aware of the fact that there are many grades and variations in the quality of wools and while we may generally class them as fine or quarter blood, defective or clear, etc., there are many variations in individual lots with respect to grease content, character of vegetable matter and extent of entanglement with fibers, and while altogether we have processed approximately 500,000 pounds of wool, any conclusions from our results should be lim-

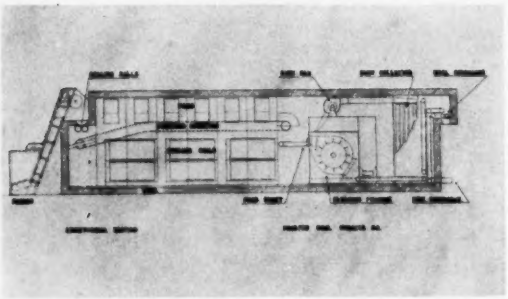


Fig. 20—Longitudinal Cross-Section of another Arrangement of Wool Freezing Chamber showing Freezing Coils below Freezing Conveyor

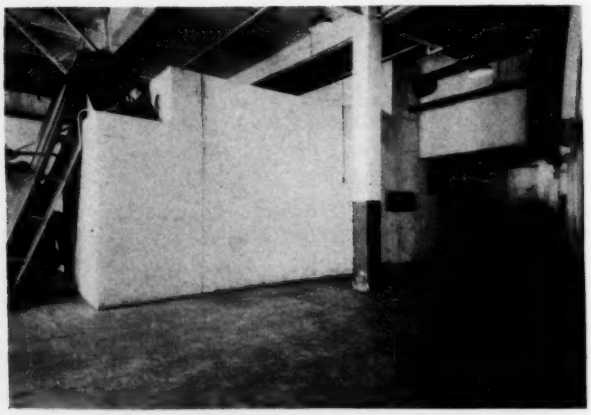


Fig. 21—Exterior View of Wool Freezing Chamber as Installed at the Lowell Textile Institute

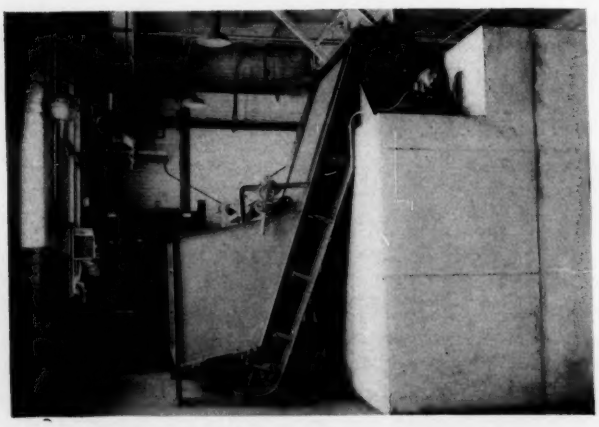


Fig. 22—Feeder for Delivering Wool to the Freezing Chamber after Sorting and without any Preliminary Opening of the Wool

ited to the particular wools we have handled and the subsequent treatment the wools received.

About 30 per cent to 80 per cent of the total normal shrinkage of the wool is removed by the freezing process. This varies, depending upin the type of wool and character of foreign matter. In some cases as much as 90% of the total shrinkage has been accomplished by the freezing process. The percentage of vegetable matter removed varies from 60 per cent to 95 per cent, averaging better than 80 per cent for defective wools. The vegetable matter removed from several typical wools is shown in Table 3. (Figure 23).

TABLE NO. 3

Vegetable Matter in Typical Wools Before and After Treating by the Frosted Wool Process

Sample No.	Description	Nature of Veg. Matter	% Vegetable Matter Based on Grease Weight of Wool Before Processing	% Vegetable Matter Based on Grease Weight of Wool After Processing
647	72s Cape	Mestiza Burrs	1.02	0.13
652	64s	Seedy and Burry	1.20	0 17
630	64s	Mestiza Burrs	2.34	0.17
651	64s	Mestiza, Wire Grass-Fileria	5.71	0.64
672	Fine 8 mo. Texas	Free Wool	0.62	0.10
666	62s	Free Wool	0.66	0.26
685	62s	Seedy, Burry, Shives	4.83	0.32
673	3/8s	Free Wool	1.72	0.36
660	3/8s	Seedy and Burry	3.18	0.43
629	3/8 to 1/4s	Seedy and Shives	3.72	0.13
680	3/8	Seedy and Burry	7.77	0.54
659	1/4	Seedy and Burry	3.60	0.23
663	1/4	Seedy and Shives	4.88	0.48
684	1/4	Seedy and Burry	6.83	0.48
665	Low	Seedy and Shives	4.00	0.49
667	Low BA	Mestiza Burrs	7.93	0.68

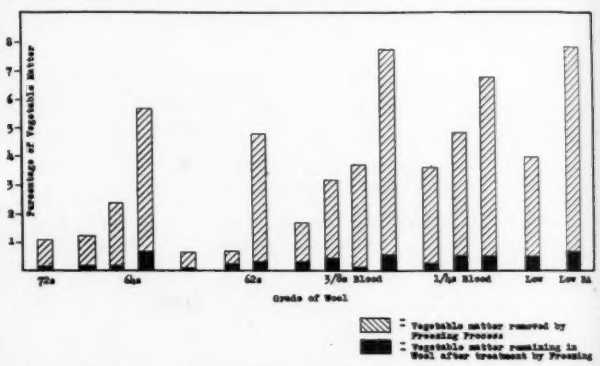

Percentage of Vegetable Matter
Grade of Wool
Vegetable matter removed by Freezing Process
Vegetable matter remaining in Wool after treatment by Freezing

Fig. 23—Vegetable Matter in Typical Wools before and after Treating by Frosted Wool Process

In Figure 24 is shown on one inch cross section paper the actual amount of vegetable matter, (over 8.70 per cent) in one-half pound of a defective domesitc quarter blood grease wool and along side the amount of vegetable matter remaining (six-tenths of one per cent) in the same half pound after treatment by the frosted process.

Figure 25 similarly shows the amount of mestiza burrs in wool before and after treatment by the frosted process, the vegetable matter in one-half pound of grease wool being reduced from 3.26 per cent to 0.27 per cent.

The effectiveness of removing the more objectional pieces of vegetable matter is shown in Figure 26.

The amount of grease removed on the dry weight of fiber expressed as keratin is shown in Table 4, (Figure 27). It varies from 30 to 60% of the total grease in the wool. The amount of grease removed by the process increases as the freezing temperature is lowered.

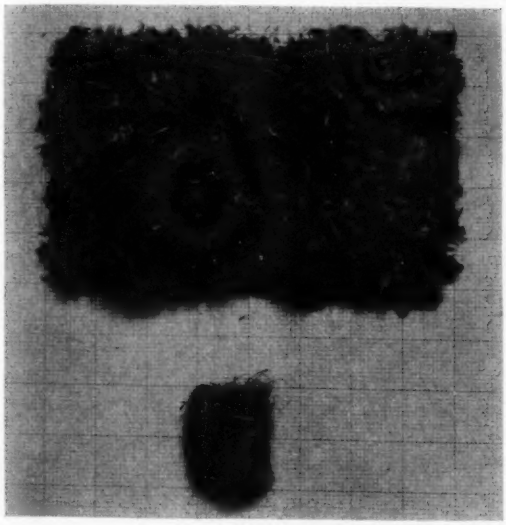


Fig. 24—The Quantity of Vegetable Matter in One-half Pound of Defective ¼ Blood Grease Wool before and after Treatment by the Frosted Wool Process

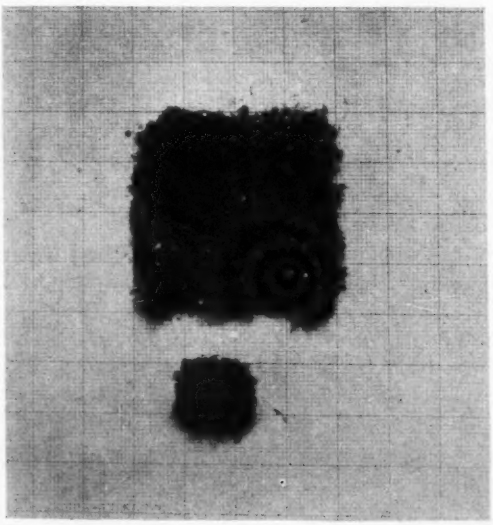


Fig. 25—The Quantity of Mestiza Burrs in One-half Pound of a Grease Wool before and after Treatment by the Frosted Wool Process

One of the difficult problems experienced with the commercial size unit, was the collection of the dust in the refrigerated chamber. We have spent many months trying to develop a satisfactory method of collecting the dust, after removing it from the wool. The serious problem is that this dust must all be collected within the limited space of the freezing chamber, for the dust must be removed from the air while still in a frozen state. The dust can not be permitted to accumulate in the freezing chamber, or it will be deposited on the freezing coils and require frequent washing of these coils.

Apparently, substantially all the moisture brought into the freezing chamber with the wool freezes in the wool

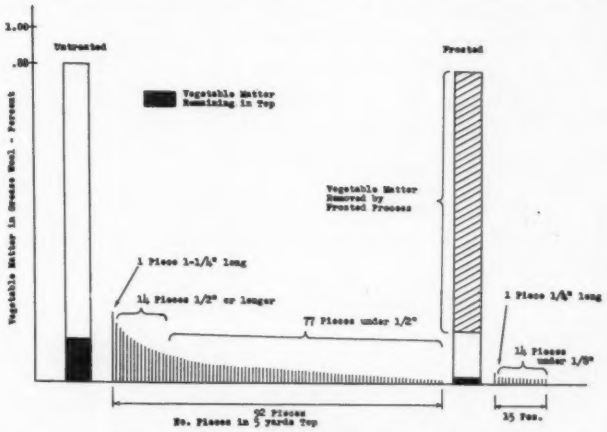


Fig. 26—Effectiveness of Removing Vegetable Matter from Wool by the Frosted Wool Process as Shown by Examination of Comparative Tops

TABLE NO. 4

Percentage of Grease Removed from Several Typical Wools by the Frosted Wool Process

(*Grease content expressed as percentage of keratin*)

Lot No.	Type of Wool	Grease Content % *Before Processing*	Grease Content % *After Processing*	% of Grease Removed by Frosted Wool Process
140	64s Calif.	37.50	26.20	30.1
200	64 Matchings	50.50	32.30	35.0
600	64s Calif.	28.90	14.60	49.4
121	⅜s Pulled	5.64	3.98	29.4
127	⅜s Calif.	20.27	8.61	57.5
220	⅜s Matchings	19.70	12.50	36.5
601	⅜ to¼ Calif.	20.00	10.10	49.5

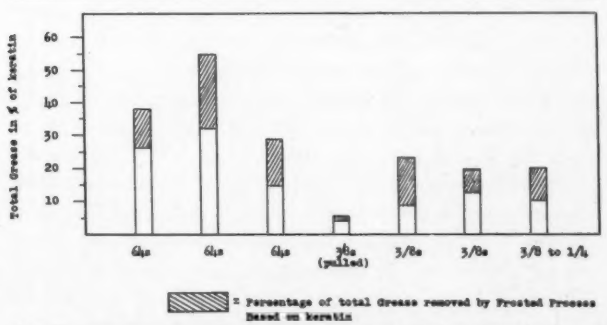


Fig. 27—Percentage of Grease Removed from several Typical Wools by Frosted Wool Process (Grease content expressed as percentage of keratin)

for very little collects on the freezing coils. Under normal commercial operation these coils may require defrosting once a month, possibly less often. We have found from our determinations that the moisture content of the Frosted Wool is less than the grease wool and the degras contained a higher percentage of moisture than does the keratin.

The frosted wool process is wholly mechanical. The total time required for the wool to pass through the process is less than three and one-half minutes, of which three minutes is required for freezing and from 5 to 10 seconds for the actual cleaning operation with wools grading from quarter blood to fine. The capacity with such wools is from 1,000 to 1,500 pounds of fleece wool per hour. Other wools may require more, or less time, depending upon impurities to be removed, and the capacity will vary accordingly.

DEFECTIVE COMBING WOOLS

The effectiveness of removing vegetable matter from defective wool by the freezing process is shown in Figure 28. A grease wool containing 5.79 per cent of vegetable matter, including mestiza burrs, was scoured, carded and combed. The vegetable matter remaining in the top was .05 per cent and in the noil was 1.16 per cent, a total of 1.21 per cent, or 21 per cent of the original vegetable matter.

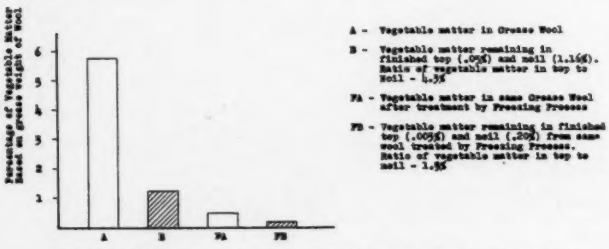

Percentage of Vegetable Matter Based on grease Weight of Wool
6
5
4
3
2
1
A
B
FA
FB
A - Vegetable matter in Grease Wool
B - Vegetable matter remaining in finished top (.05%) and noil (1.16%). Ratio of vegetable matter in top to Noil - 4.3%
FA - Vegetable matter in same Grease Wool after treatment by Freezing Process
FB - Vegetable matter remaining in finished top (.003%) and noil (.20%) from same wool treated by Freezing Process. Ratio of vegetable matter in top to noil - 1.5%

Fig. 28—Effectiveness of Removing Vegetable Matter by Freezing Process

Another lot of the same grease wool, after treating by the frosted process, contained before scouring only 0.49 per cent of vegetable matter on the original grease weight and after scouring, carding and combing the vegetable matter remaining in the top was 0.003 per cent and in the noil was 0.20 per cent, or a total of 0.20 per cent of the original grease weight, or 3.5 per cent of the original vegetable matter as compared with 21 per cent for the untreated wool. Furthermore, the ratio of vegetable matter in top to noil for the untreated wool was 4.3 per cent and for the frosted wool 1.5 per cent, which shows the remaining vegetable matter is more easily removed from Frosted Wool by combing. By count there were 29 pieces of vegetable matter in the frosted top as compared with 217 pieces in the untreated top per five yard length.

The above results are not exceptional but are representative of the results obtained with most badly defective wools. Shives and seeds are more easily detached and removed than are mestiza burrs.

There have been some defective wools which we have not as yet been able to clean effectively without some shortening of the staple and also wools containing extremely small mestiza burrs, or wools with very low grease content, that we have not been entirely successful in handling. Further study and testing will be necessary with these wools.

PROCESS SUCCESS FOR DEFECTIVE WOOLS

While we cannot guarantee that satisfactory results will be obtained with all wools, the frosted wool process has been demonstrated as satisfactory for the removal of the usual forms of vegetable matter from most defective combing and clothing wools of ¼ blood to fine grades, making these wools equal or superior in quality to free wools of the same grade and staple length as the Frosted Wool. After the frosted treatment, these defective wools may be combed alone or mixed with the free sort and subjected to the normal processing for worsted or clothing wools, which is a procedure that is not desirable with carbonized wools.

The processed fiber of frosted defective wool is usually more free of vegetable matter than the normally processed clear sort of the same wool.

The frosted processed wool makes a whiter, loftier top and noil. The individual fiber strength is preserved although the length of staple may be slightly shortened as compared with the free sort fleece wool. The yield of usable fiber is greater than that obtained from the carbonizing process.

PAINT SORTS, TAGS AND OTHER OFF SORTS

A number of different off sorts of wool have been treated by the process.

It has been found that oil-paint and asphalt-paint can be effectively removed from paint sorts by the freezing process. The paint becomes brittle upon freezing, much in the same manner as the grease of the wool, and flakes off of the fibers in the cleaning machine. Unusually large hard lumps are probably broken off the wool.

Usually more time is required in the cleaning machine for the depainting operation than for processing ordinary wool and therefore the capacity may be reduced below 1,000 pounds per hour with the present equipment.

The wool depainted by the frosted process is not absolutely free of all specks of paint but any specks that may remain are usually very small and completely detached from the wool and drop out in carding. Several lots of wool of several thousand pounds each that were depainted were mixed back with the regular free sort for manufacturing into worsted yarn. Tags have also been effectively opened and cleaned by the frosted process without preliminary preparation.

Card strippings and card wastes containing oil have been successfully cleaned.

Heavily cotted wool has been thoroughly and effectively opened without knotting.

The process as now operated will not remove vegetable matter from noils as taken from the comb.

FROSTED PROCESS AND FREE CLEAN WOOLS

Very complete tests are being made in a number of mills with wools treated by the frosted process to determine the effect of the freezing process on all the properties of the wool as developed under commercial processing conditions. A summary of the results of some of these tests and investigations will be available in the near future. Much time and expense is necessary for this work and a number of lots of wool must be processed in each case in order to secure reliable information, because of the many separate handlings such test lots must receive with possible errors in a commercial plant.

Our work on so called free wools has not yet been carried to a conclusion. Many thousands of pounds of free wools have been treated, some of them successfully without shortening of staple, nor increase in noilage, nor loss of yield. Also the value of the freezing process has been indicated in a resultant cleaner top and noil with greater whiteness and bloom and all the incidental economies resulting from starting the normal worsted processing with a cleaner wool in the scouring bowl.

Some of these commercial tests on free wool, however, have not been equally satisfactory and the reason for this has been two fold. First, the treatment which the wool is given by the frosted wool process in the cleaning machine may be controlled so that it is little more severe than an opening operation, or it may be made very extensive and cause more or less incidental breakage and shortening of fiber. The severity of the treatment is entirely under the control of the operator and will be determined by the result he wishes to accomplish. The frosted processing may be varied over a wide range. Every wool has slightly different characteristics and this requires considerable conjecture as to the proper setting of the machine and the correctness of the treatment can only be determined after the frosting process is finished and the wool has been returned to the mill for processing, and several days or weeks elapse before the mill results are available. Our men are gradually becoming more skilled in handling these free wools and recent results have been very encouraging.

It is recognized, however, that truly comparable results with normal mill practice on free wools cannot be obtained until the frosted process is installed in a mill where the process can be made continuous with the regular mill operation, with the normal hourly checking on performance which is only available under such conditions.

Second, Frosted Wool is in a more open lofty condition, which requires some slight variation in the setting of machines and in methods of subsequent processing to get the best results.

Figure 29 shows how the vegetable matter is removed by the normal process and the frosted process from a free wool cotaining only .98 per cent of vegetable matter on

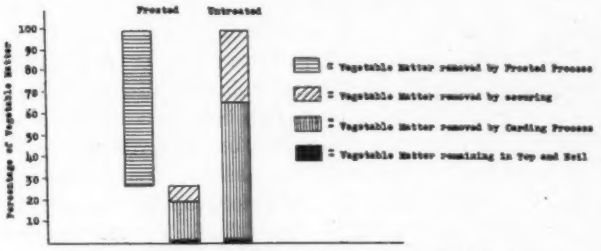


Fig. 29—Removal of Vegetable Matter from a Free Wool (Vegetable matter = .98% of grease weight)

the grease weight of the wool. The top made from the normally processed wool contained 41 pieces of vegetable matter in a five yard length, while the Frosted Wool contained 10 pieces in a similar length.

We have done less work with clothing wools as distinct from worsted wools, but there is no reason why the work on combing wools should not be largely applicable to clothing wools.

Some preliminary work has been done on carpet wools and from these tests it appears that they can be as effectively cleaned as the combing and clothing wools, but some changes will need to be made in the equipment for handling these longer wools and this work must be postponed until the present demonstrations are completed.

We perhaps have set our objective a little high in attempting to handle the so called free clean wools through our process before we have had several of these units in continuous commercial operation on defective wools, but through the encouragement of the executives of several of the large wool companies we have been employing a staff of from 10 to 20 engineers and technical men who are devoting their time to the development of the process and present results make us hopeful that the process will in a reasonably short time be successfully applied to all wools.

COST OF PROCESS

The cost of one complete unit, installed in an existing wool plant, ready for operation, with a capacity of 1,000 to 1,500 pounds per hour of fleece wool, including both refrigerating and processing equipment, will be between $35,000 and $40,000, of which amount approximately half is for refrigerating equipment and the remainder for the cork room and wool processing equipment. If several units are installed at one time the per unit cost would be somewhat less.

The actual operating cost with power costing 1.5 cents per kilowatt hour, including depreciation and all direct costs, but excluding royalty, is less than one-half cent per pound of grease wool treated.

ADVANTAGES OF FROSTED WOOL PROCESS

The following summary of the application of our results must be considered tentative for we realize there are many wools with which we have had only meager experience.

By use of the frosted process, all mill processing operations will be started with more free, clean and open stock.

The frosted process will produce a cleaner, more resilient, lofty, superior quality top, noil and finished product.

Lower cost defective wools may be purchased and used in products where heretofore their use has been impossible or uneconomical because there has been no method of cleaning such wools without deteriorating the wool fibers.

No carbonizing of virgin wools should be necessary except for white stock and possibly some extraordinary wools.

The lower cost of the frosted process will result in material savings in the cost of cleaning vegetable matter from defective wools in addition to furnishing a materially superior product.

There is a saving in scouring expense because of the reduction in time required and partial, or entire, elimination of the need of alkali and reduction in soap.

There will be many incidental savings such as reduced cost due to added capacity of dryers, because of the more open, fluffy condition of wool, a reduction of operating and maintenance cost in carding, longer life of card clothing and greater use of metallic clothing, lower maintenance cost of combs, incidental savings in specking and mending, savings in piece carbonizing, reduction in probability of damage from paint in the finished product and savings in bleaching and dyeing expense.

To some mills the reduction in stream pollution will be of importance.

A NEW FACILITY FOR WOOL PROCESSING

The process should be considered as a new tool in the hands of the manufacturer to be used as he finds by experience to be best suited for the particular wools and results he is trying to obtain.

In general, the use of the frosted wool process should result in an improvement in quality of finished goods and lower overall production costs, although it may require a reasonable period of mill operation to fully develop all of its possibilities.

DISCUSSION

Question: I wonder if the freezing process removes paint?

Answer: Yes. It will not remove a stain, but it will remove hard paints, such as asphalt, oil and tar paints.

Question: At what temperature do you operate?

Answer: The temperature at which we are operating is between 25° and 35° F. below zero. There is no harm in using a lower temperature, atlhough the expense is greater.

Question: How about the time?

Answer: The total time required for the entire processing varies from three to five minutes, depending upon the time required to freeze the wool, which varies with the density of the grease. The actual processing time after freezing is only a few seconds.

Question: Have you had any experience with New Zealand wool with about one per cent burrs in it?

Answer: No. But we have with Australian wool containing from 3 per cent to 5 per cent of Mestiza burrs. The Australian wools containing Mestiza burrs, which we processed, had previously been subjected to considerable pressure in the baling so the burrs had been mashed into the grease, thoroughly entangled with the fiber and more or less broken, so we were unable to do an entirely satisfactory job with the small lots we have had of very burry Australian wool. We have successfully removed large quantities of Mestiza burrs from domestic wools and on some of the Australian wools. We have successfully removed every type of vegetable matter received in defective wools without exception.

Question: I don't get the purpose of the condenser.

Answer: The condenser is an ordinary type of wool condenser, which is commonly employed in textile mills for collecting fibrous matter from an air stream. In our case, the wool is condensed so it can be discharged from the room through the sealing rolls without the air which must be retained in the room.

Question: Could you take hold of the wool as it comes out of the freezing equipment?

Answer: Yes. It feels a little cold just for a few seconds, but not uncomfortably so.

Question: How much moisture is there in it?

Answer: It depends on the humidity at the time the wool is processed. The normal amount of moisture in the wool as it enters the freezing process may vary from 8 to 10 per cent and as the wool leaves the freezing chamber, the moisture content of the wool would be one or two per cent less, as the dirt, vegetable matter and grease which is removed from the wool carries off a higher percentage of the total moisture originally in the grease wool. The moisture content of the wool just before processing is unimportant for the process relies on the freezing of the grease. After processing the grease wool, it can be immediately handled, stored, shipped or scoured without further treatment. A scoured wool can be treated by another process we control which requires wetting the wool, but it is more expensive. If the scoured wool is treated by moistening and freezing, it will contain around 25 to 28 per cent moisture when discharged from the freezing chamber, which means it will have to be immediately processed or dried if it is to be stored.

Question: Is there anything circulating between the cleaning device and the freezing chamber?

Answer: No, except the air from the cleaning device, after having the dust removed from it, is recirculated through the freezing coils. The atmosphere around the

cleaning machine is substantially as cold, within a few degrees, as it is under the freezing coils. The air in the entire freezing chamber is recirculated through the freezing coils many times a minute.

Question: Do you strain that air and use it over again?

Answer: Yes, as stated previously, the air is continuously recirculated in the chamber where the freezing conveyor carrying the wool passes, just as it does in any wool drier. This air does not pick up dirt from the wool so it is not necessary to clean it as it is recirculated, but the air from the cleaning machine does carry large quantities of dirt and that must be filtered before recirculating. The removal of dirt from the air is done by passing it through a bag filter and there the dust is filtered out, in the same manner as in a vacuum cleaner. Since the dirt and grease is dry and hard, a bag filter can be used continuously by occasional shaking, which is done automatically.

Question: Do you find a difference in the breaking strength of the yarn made from the wool?

Answer: Yes. We have made many tests at the Institute and there have been many tests made by the mills. These tests have shown in some cases higher breaking strengths for yarn made from Frosted Wool and in other cases the strength has been lower or the same as the untreated wool, but in no case has the difference been great. In practically all cases the Frosted Wool has shown uniformity of strength and greater elasticity than the untreated wool.

Question: How does it compare with the wool carbonized by a baking process?

Answer: I can't answer that question. It is my understanding no worsted yarns are made from straight carbonized wool.

Question: If the wool is carbonized straight, how does the breaking strength of the yarn compare with wool that is carbonized by your freezing process?

Answer: Wool is not carbonized by the freezing process. The fiber itself is not damaged, or changed in any way by the freezing process, unless it is very badly contaminated with vegetable matter or tar, in which case it must be retained longer in the cleaning machine and the fibers may be somewhat shortened. The fibers are not dehydrated or weakened, as I understand is the case with fiber which has been exposed to carbonizing.

Question: Have you ever processed any shoddy material that contains cotton or rayon?

Answer: The freezing process cannot be used for removing vegetable matter from shoddy if the shoddy does not contain grease or oil which can be frozen. It is possible the wet freezing process, which I have previously referred to, could be used for removing vegetable matter from shoddy but we have not experimented with it and I would not venture to guess as to the possibility of successfully treating shoddy in this manner.

Question: Would you care to say how much money has been spent in this process?

Answer: Entirely too much. We have been working on it for approximately three years. It has required the expenditure of a large amount of money, as do all worthwhile pioneering ventures of this type. While the original idea contemplated the use of the process for defective wools only, through the encouragement from the mills, we are now applying the process to the so-called free or clear wools. Even the best of wools carry an appreciable amount of vegetable matter, all of which is costly to remove by the present processing methods. Perhaps few mills have recognized the great expense this small amount of vegetable matter has caused them because they have become accustomed to working with such wools. The Frosted process is logical in that by this method the vegetable matter is removed before any of the normal subsequent manufacturing processes are started.

Question: How is the affinity for dyestuff of ordinary wool as compared with the Frosted Wool?

Answer: At the Lowell Textile Institute we made a large number of tests with a number of different typical dyes, using a number of different types of wool with duplicate samples of frosted and untreated wool and from these tests concluded there was practically no difference in the manner in which the Frosted Wool absorbed the dye. In general, the Frosted Wool is whiter after scouring and in some of the mill tests it has been found that the dyed stock is brighter with the same amount of dye, which would be expected from a whiter stock.

Question: Pertaining to the grease in the wool process, is the scouring modified? Do you use four or five bowls?

Answer: The scouring process varies with the different mills but in every case they have found very much less soap is required and in many cases no alkali is used with the frosted wool. This practice varies, however, and we have not attempted to make any recommendation to the mill with respect to scouring, other than to caution them that the frosted wool may be easily overscoured. The wool stock is so open and the small amount of grease remaining on the fiber so free, that very little scouring is required. Three bowls are sufficient.

Question: What percentage of grease is removed?

Answer: We normally remove from 30 per cent to 70 per cent of the grease in the orginal grease wool.

Question: What are the prospects of applying this to woven pieces instead of the raw stock?

Answer: I cannot see at present how the process could be successfully applied to woven goods, although it is possible by wetting the goods the freezing method might be made applicable.

Question: The wool ordinarily is steeped in its own soluble dirt whereas in your process this material is removed from the wool in dried condition so that the subsequent steeping of the wool is not in its own dirty matter.

Answer: You are correct—the scouring liquor would be relatively very clean and therefore much of the soluble matter that might stain the wool is not present in the scouring bowl.

Question: Have you tried any pulled wool?

Answer: Yes, and we have successfully treated by the Frosted process pulled wools containing 4 per cent or 5 per cent of grease.

Question: How much lime was removed?

Answer: I cannot answer that question accurately. We know that most of the lime was removed, but we have made no accurate determinations.

Question: How can you tell after it runs through the machine whether it is right or not before it goes to the scouring bowl?

Answer: We can only tell by experience we have had with similar wools. If it is a new wool, which we have not previously processed, we must run a preliminary lot and check with the mill. We can hand-scour samples to get some idea, but we cannot know accurately without having the mill process the wool after we have treated it. Normaily we like to have several thousand pounds of wool for a test, as the machine has a capacity of 1,000 pounds or more per hour and it is desirable to have several hours run as a minimum in order to intelligently handle the wool, unless we have had previous experience with the same wool.

Question: Have you tried it on mohair?

Answer: We have not made an adequate trial of mohair. Some small samples have been processed but the quantity was not sufficient to tell the story.

Question: Does the felting properties of the frosted wool equal those of the regular processed wool?

Answer: Yes. From all the experience we have had, the felting quality of the wool is improved rather than deteriorated. Scales on the fiber are not affected by our process. We have examined many samples of all grades of wool under the microscope and our conclusions have been verified by Professor Schwarz of the Massachusetts Institute of Technology, who has also made a comparison of the frosted and untreated fibers on a number of grades of wool.

Question: Will it remove string?

Answer: No, it will not.

Question: Do you carbonize the noils?

Answer: If the frosted process has done a good job, the noils are several grades better than would normally be obtained and for most uses would not need to be carbonized. Such vegetable matter as does remain in the noil usually is very short in length so that there is a much greater probability of it dropping out in the subsequent manufacturing processing.

Question: If you try to freeze the wool dry, does that show any material difference?

Answer: Your question is not clear; but if we put a scoured dry wool containing vegetable matter in our freezing machine, will the vegetable matter be removed? The answer is "No," unless there is grease or a reasonable amount of oil or water present in the wool—the freezing process will not remove the vegetable matter. It is possible by your question you are asking if scoured wool can be dried by freezing?

Questioner: Yes.

Answer: The wool can be dried in the freezing chamber by blowing air through the wool for an appreciable length of time. However, from the small amount of work we have done, it would appear the cost would be high and such a method would not prove economical.

Question: What is the relationship between the grease content and the burr removal?

Answer: There is no direct relationship, although it seems to be easier to remove vegetable matter from greasy wools, unless the grease is in the form of hard lumps, in which case the freezing must be prolonged in order to make this grease completely hard and brittle so that it will shatter. It is possible that some greases freeze at higher temperatures than others, but the temperature that we are using appears to be low enough to freeze all greases that we have found on the wools we handle.

Question: You would say that the removal of burrs is dependent on the removal of the grease?

Answer: Yes. The effective removal of the burrs depends upon the freezing of the grease in the wool. The most difficult form of vegetable matter to remove is the mestiza burr.

Question: What happens to the wool fiber itself that produces more short fibers?

Answer: If any shortening of the staple is found in the wool which has been processed by the freezing method, the shortening is due to a mechanical breakage of the fiber and not to shrinkage. There is no chemical or physical change in the fiber itself.

Question: How do you account for the less noils in the carding?

Answer: Where less noils have been obtained on the frosted wool than on the untreated wool, this is probably due to the fact that the frosted wool is more open when it enters the scouring bowl and therefore does not become so matted during the scouring and drying process and does not receive so much tearing action on the cards during the carding process. Generally the difference in the percentage of noils of the treated and untreated wools is not great and may be within the limit of variation which would be found in testing such variable raw material.

Question: Does the lower temperature cause more fiber breakage?

Answer: No. The freezing itself causes no breakage of the fiber.

At the conclusion of the discussion, Mr. Wig informed the meeting that his paper was the first public statement about the Freezing Process. He expressed his pleasure at the manner in which the subject was received and extended an invitation to anyone interested to examine the equipment at the Lowell Textile Institute, but suggested that this be done by appointment.

APRIL MEETING, PHILADELPHIA SECTION

THE Philadelphia Section met in the Broadwood Hotel, Philadelphia, on Friday, April 26th, with 55 present.

Norman D. Harvey Jr. of Carbide and Carbon Chemicals Corporation delivered a paper on, "Aliphatic Organic Chemicals in Textile Processing".

It was announced that the Annual Outing would be at the Torresdale Country Club on June 7th.

Respectfully submitted,

R. W. DELANEY,
Ass't Secretary.

MEETING, NORTH CAROLINA STATE COLLEGE STUDENT SECTION

THE North Carolina Student Section had as their speaker the week of May 4, George Searell of the Jacques Wolf Company.

Mr. Searell gave two talks in one of which he explained the various processes in silk throwing; in the other he told briefly how and why strippers were used. He also gave some interesting data on the use of enzymes as used in desizing.

The senior class in Textile Chemistry and Dyeing was the guests of Mr. Searell at a dinner in the evening.

Respectfully submitted,

A. E. SHUMATE,
Secretary.

SPRING MEETING, SOUTH CENTRAL SECTION

THE South Central Section held its regular Spring meeting at the Read House in Chattanooga, Tenn., on April 27, 1935. Thirty-two members and guests were present for dinner and an especially entertaining program.

The minutes of the last meeting were read and approved.

G. B. DuBois was re-elected councilor. Walter E. Hadley was named for the nominating committee.

It was voted to again hold the Summer meeting on Signal Mountain in Chattanooga and extend invitations to the Piedmont and Southern Sections.

Chairman Harold Schroeder introduced the speaker for the evening, John E. Robertson of the duPont Co. His paper on peroxide bleaching was greatly appreciated and a lengthy and constructive discussion followed. A rising vote of thanks was given Mr. Robertson.

Respectfully submitted,

H. B. KIMBRELL,
Secretary.

MEETINGS, LOWELL TEXTILE INSTITUTE STUDENT SECTION

A MEETING of the student section of Lowell Textile Institute was held May 2, 1935. Edward S. Chapin, the guest speaker, gave an interesting talk on "Ageing of Printed Textiles". The meeting was well attended by members and guests.

On May 10, 1935, another meeting was held for the purpose of electing officers. Herbert A. Wormwood of North Wilmington was elected Chairman and Henry S. Anthony of Lowell was elected Secretary.

Respectfully submitted,

CHESTER M. KOPATCH.

Proceedings of the American Association of Textile Chemists and Colorists

Active Ingredients and Total Fatty Matter in Sulfonated and Sulfated Oils

Committee Report No. 4

By RALPH HART
*Chairman, Sub-Committee on Sulfonated Oils**

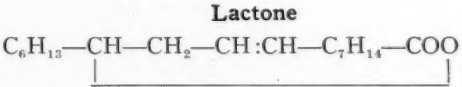
THE test for fatty matter is considered the most important determination in the analysis of sulfonated oils, primarily because the price of the oil is based upon it. This determination has consequently been the subject of considerable investigation for a good many years with the result that a number of methods have been proposed, many of which are in use but unfortunately do not always yield concordant results. In response to a questionnaire addressed to mill and commercial laboratories and to manufacturers of sulfonated oils, Grimshaw[6] reports as many as 25 methods in use, giving results which in some cases differed by more than 10 per cent. Fahrion[4] analyzed a sample of sulfonated castor oil by three accepted methods and found that the results varied from 78.2 to 82.9 per cent, or a difference of 4.7 per cent. This committee has investigated several of the more common methods and also a new proposed method, called the total active ingredients method. It is considered that the new method yields not only the *true* fatty matter but is also applicable to true sulfonated fatty and mineral oils, i.e., oils which are not decomposed by boiling with mineral acid—in which cases the usual methods are useless.

COMPOSITION OF SULFONATED CASTOR OIL

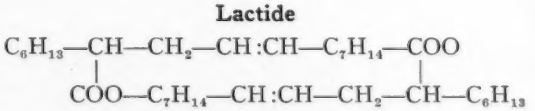
A study of the probable constitution of sulfonated oils before and after decomposition, as reported in the literature, is of considerable help in investigating methods for their analysis. Compared with other oils or fats that are commercially sulfonated, the composition of sulfonated castor oil is the most complicated, due to the tendency of its fatty acids to split off water with the formation of polymers and condensation products. Some of the polymers of castor oil fatty acids, the latter consisting mainly of ricinoleic acid, most frequently discussed in connection with sulfonated castor oil are:

Ricinoleic Acid

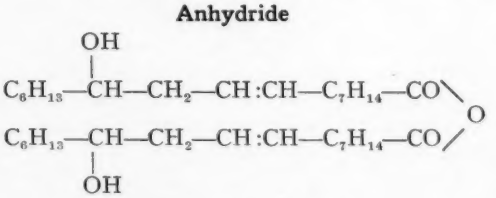
Lactone



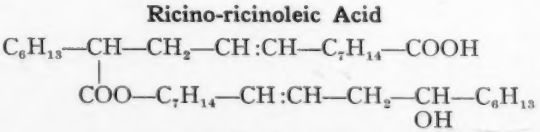
Lactide



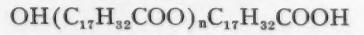
Anhydride



Ricino-ricinoleic Acid



Polyricinoleic Acid



**Members of the sub-committee on sulfonated oils*:

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Fahrion[5] considers that the principal reactions that take place between concentrated sulfuric acid and ricinoleic acid under the usual manufacturing conditions are the esterification of the hydroxyl group—yielding the sulfuric acid ester—and the dehydration of the fatty acids to form polyricinoleic acids, of which ricino-ricinoleic acid is the first member. The formation of anhydrides, lactones, or lactides is considered negligible. He is also of the opinion that sulfonated castor oil consists of the sulfonated fatty acids and is practically free of sulfonated glycerides, as the latter even if formed during the reactions are very unstable. Winokuti[14], Richardson and Walton[13], and many others agree with these conclusions. Richardson and Walton believe, however, that some sulfated monoglyceride, which is easily decomposed, may be present. They further state that the sulfate of the simple fatty acids only are formed and that sulfated polymerides are probably not present. The approximate composition of a commercial sample of sulfonated castor oil, according to Richardson & Walton, is given in Table I.

TABLE I
Approximate Composition of Castor Turkey Red Oil per 100 Parts Decomposed Fatty Matter

	%
Unsaponifiable	0.42
Diricin olein	10.52
Triricin olein	2.63
Ricino-ricinoleic acid	8.33
Ricinoleic acid	47.50
Sodium sulfate sodium ricinoleic acid	36.83
Sodium sulfate monoglyceride ricinoleic acid	11.00
Free glycerine	2.06
Glycerine in easily hydrolysable esters	2.14
Glycerine in stable glycerides	2.03
Total glycerine	6.23
Combined SO_3	8.83

COMPOSITION OF DE-SULFATED OIL

In the usual methods for estimating total fatty matter in sulfonated oils, the sample is decomposed by boiling with mineral acid and the extracted fatty matter, which then may or may not be saponified, is finally heated in an oven at about 105° C. to constant weight. It is obviously of importance to follow through the changes in the fatty matter, particularly in so far as it affects its weight, as a result of the various steps in the analytical procedures. According to Herbig[9], as much as 10 per cent of the glycerides in a sample of sulfonated olive oil hydrolyzed upon boiling with dilute hydrochloric acid; even the raw oil decomposed to the extent of about 3 per cent when boiled for 1 hour with the same strength acid. The present writer boiled a 10-gram sample of a concentrated sulfonated castor oil with 35 cc. of N sulfuric acid for 1 hour and found that the acidity of the fatty matter increased from 117.5 to 133.0 mg., expressed as KOH per gram of anhydrous fatty matter, or an increase of 15.5 mg.—indicating considerable decomposition of the glycerides. The increase in acidity is undoubtedly due to hydrolysis of the glycerides, since hydrolysis of the polymers probably does not occur in acid solutions. In fact the reverse seems to take place, i.e., polymerization and condensation products of the fatty acids are formed with a corresponding loss in acidity. Hence the loss in glycerine was undoubtedly even greater than indicated by the apparent increase in acidity.

An entirely different result was obtained upon boiling the sample for 1 hour with 50 cc. of strong hydrochloric acid (sp. gr. 1.19). Instead of an increase in acidity there was a drop—namely, from 117.5 to 96.1 mg., or a loss of 21.4 mg. The loss in acidity can be due only to a polymerizing reaction in which water is eliminated. It is obvious that the polymerization must have been greater than indicated by the loss in acidity, since some hydrolysis of the glycerides had undoubtedly taken place at the same time. The polymerization of ricinoleic acid upon boiling with mineral acids is well known and often mentioned in the literature. Thus Winokuti[14] reports that, upon boiling with approximately 2N hydrochloric acid for 5 hours, the acidity of the fatty matter obtained by acid decomposition of a sulfonated castor oil fatty acids dropped from 172.3 to 158.8 mg.

Polymerization also takes place upon drying the fatty acids at high temperatures. Winokuti[14], for example, reports a loss of acidity of almost 50 per cent upon heating a sample of ricinoleic acid at 105° to 110° C. for 70 hours. His other data on this point are given in Table II.

TABLE II
Effect on the Acidity of Castor Oil Fatty Acids upon Heating at 105° to 110°C.

Time, hours	*Acidity mg. KOH per gram of fat*
0	187.5
5	173.5
20	138.7
30	124.7
40	108.5
50	105.9
60	100.3
70	96.1

The present writer, upon drying a sample of ricinoleic acid for 2 hours at 105° to 110° C., obtained a drop of 4.2 mg., as KOH per gram of fat, which in the ordinary analysis is practically negligible.

According to Rassow-Rubinsky[12] and Winokuti[14], upon

heating ricinoleic acid, only ricino-ricinoleic and its homologues are formed but no lactones, lactides or anhydrides, or at least not to any great extent. Gruen and Waldenberg[7] claim that upon continued boiling, sulfo-ricinoleic acid is converted into the lactide. This is confirmed by Richardson and Walton[13] who further state that ricinoleic acid is also formed and that the longer the boiling the greater the amount of the lactide. This is disputed by Winokuti[14] who, after boiling a sample of sulfo-ricinoleic acid with 2N hydrochloric acid for 5 hours, found only a trace of neutral bodies. The writer believes that Winokuti's method of extraction may have been at fault.

At any rate as far as the analysis is concerned, the nature of the polymers is of no consequence, since in all cases the loss in acidity of one carboxyl group corresponds to the loss in weight of one molecule of water. It has already been mentioned that upon boiling a sample of sulfonated castor oil with dilute hydrochloric acid, there was an increase in acidity of 15.5 mg. and with strong hydrochloric acid, a drop of 21.4 mg. The former corresponds to a loss of the glyceryl radical of about 0.4 per cent, due to the hydrolysis of the glycerides, and the latter a loss of water of about 0.7 per cent, due to polymerization of the fatty acids. These losses are minimum values, since they only take into account either hydrolysis or polymerization, whereas both reactions occur simultaneously. It also does not consider the presence of di- and monoglycerides, which would further increase the loss of glycerine. As a matter of fact, the fatty matter determined by decomposition with dilute hydrochloric acid method showed a loss of 1.5 per cent and with strong acid, a loss of 3.3 per cent, when compared with the total active ingredients method. It is thus evident that the fatty matter determined by acid decomposition methods is bound to be low—the results varying somewhat with the strength of acid, time of heating, etc.

In the official method of the Wissenschaftliche Zentralstelle für Öl und Fettforschung (Wizoeff)[15], the sample is boiled for over an hour with 50 cc. of strong hydrochloric acid, the fatty matter extracted and saponified with alcoholic potassium hydroxide, and finally the fatty acids are determined in the soap solution in the usual way. A number of investigations[11] have shown that sulfonated castor oil as well as its acid-decomposed fatty matter may be completely saponified, including the polymers. Saponification of the fatty matter must necessarily be accompanied by loss of glycerine from the glycerides and a gain of water due to the splitting of the polymers. Hence results by saponification methods may or may not correspond to the true weight of fatty matter in the original sample, depending upon whether or not the loss of glycerine is balanced by the gain of water absorbed by the polymers.

HERBIG OR ACID DECOMPOSITION METHOD

In the original Herbig[8] method, which is similar to the method specified in Commercial Standard CS43-32[3], the sample is boiled with strong hydrochloric acid (sp. gr. 1.19) for 15 minutes, the fat extracted, the solvent evaporated, and the residue dried at 105° to 110° C. It has already been shown that upon boiling sulfonated oils with mineral acids, hydrolysis of the glycerides as well as polymerization of the fatty acids occur, both tending to diminish the weight of the extracted fatty matter. In our work it has been found that boiling for 15 minutes was insufficient to completely decompose the oil since both layers were still turbid; also that decomposition with strong acid gave lower results than with dilute acid. Obviously the lower results must necessarily be the less accurate. Committee work on this method, using N sulfuric acid, indicates that the method is satisfactory for routine analysis. The procedure in detail is given at the end of this report and is recommended as an alternative method.

THE A. L. C. A. METHOD

According to this method[1], the percentage of fatty matter is given by the difference between 100 and the sum of moisture, unsaponifiable, ammonia, sodium as soap, neutralized combined SO_3, and total salts and impurities in the oil. Evidently a complete analysis of the sample is required in order to arrive at the fatty matter. As free glycerine and other impurities are rarely determined, the fatty matter by difference is usually high.

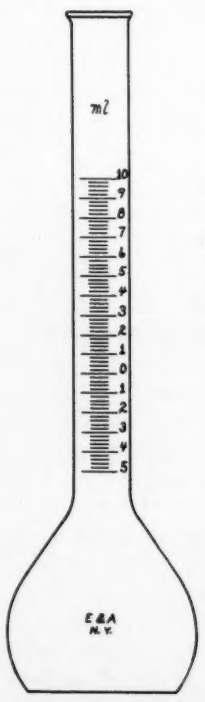

Fig. 1—Buchner Flask
Drawing, Courtesy Eimer & Amend

THE VOLUMETRIC[15] OR APPROXIMATE METHOD

Ten grams of high-percentage or 20 grams of low-percentage Turkey red oil are weighed into a 100-cc. beaker, heated with 25 cc. of water till dissolved, and by repeated washing transferred into a 200-cc. Buchner flask (Fig. 1) for fatty acid estimation, the neck being graduated in tenths of cc. Fifty cc. of concentrated hydrochloric acid are then added (sp. gr. 1.19), the mixture is heated over a small flame for at least an hour till the fat has completely separated out, and then filled up with hot concentrated sodium chloride solution till the fat has risen into the graduated neck of the flask. The flask is placed in a boiling water bath and the fat volume read off after a quarter of an hour. It is again read after another ten minutes and, if this reading is dif-

ferent from the first, a third reading is made an another ten minutes and so on until concordant results are obtained. The number of cc. of fat multiplied by the sp. gr. at 90° C. gives the weight of the fat in the Turkey red oil, from which the percentage is easily calculated.

This method is subject to the following errors or inconveniences: (a) error in reading the meniscus, (b) presence of moisture in the fat, (c) inconvenience of determining the specific gravity of the decomposed fat, and (d) errors inherent in all methods depending upon the determination of fatty matter by hydrolysis and decomposition with acid.

THE WIZOEFF[15] OR FATTY ACIDS METHOD

If the fatty acid content of the oil under test is approximately known, 8 grams in the case of 50 per cent oils, or 4 grams in the case of 100 per cent oils, are weighed out for analysis. If fatty acid content is not known, 6 to 8 grams are dissolved in 25 cc. of distilled water, 50 cc. of concentrated hydrochloric acid (sp. gr. 1.19) are added, and the mixture is heated in an extraction flask with reflux condenser until the fat has entirely separated, in any case for an hour at least. After cooling, it is transferred, together with a little distilled water and ether, into a separating funnel and when the two layers have separated, the clear acid water is removed and shaken up twice with 25 cc. of ether. The two ether solutions are washed free from acid by repeated shaking up with 20 cc. of water. If emulsions form or fatty particles separate out again when the wash water is acidulated with hydrochloric acid, the product under test still contains some sulfo acids and cannot be tested under the scheme being described. The ether solution is transferred to an extraction flask, the ether distilled off over the water-bath and the fatty residue treated with 50 cc. of N alcoholic potassium hydroxide for half-an-hour. From the alcoholic soap solution thus obtained the greater part of the alcohol (about 30 cc.) is evaporated. The residue is dissolved in 50 cc. of distilled water, acidified with 65 cc. of N hydrochloric acid, and decomposed by frequent stirring and heating for about ten minutes at 50° C. Longer heating at a higher temperature must be avoided. The separated fatty acids, after cooling, are quantitatively transferred with ether into a separating funnel. The acid water is again twice shaken up with ether (25 cc.) in order to secure the complete extraction of the fatty acids, and the combined ether extracts are washed free from mineral acid by frequent shaking with 20 cc. of distilled water, using the methyl orange test. The ether solution is now dried by frequent shaking for about an hour with 5 grams of anhydrous sodium sulfate and filtered through a dry filter into a wide-necked weighed flask. The sodium sulfate and filter are washed with absolute ether, the solvent is distilled off, and the residue blown a few times with hand-bellows in order to completely remove the last trace of solvent. It is now dried for an hour at 100° to 105° C., cooled and weighed.

Burton and Robertshaw[2] consider this method inaccurate in the following respects: (a) It may give low results due to condensation, polymerization, and sometimes "carbonization" of the free fatty acids; (b) stable sulfonic compounds are not determined; (c) some of the highly oxidized, polymerized, or degradation products are insoluble in ether and are therefore not estimated, and (d) any glycerides, or other compounds which are not hydrolized, saponified, or decomposed will be included.

TOTAL ACTIVE INGREDIENTS METHOD

The total active ingredients method, suggested by the writer, was originally developed by the A. A. T. C. C. committee on sulfonated oils in connection with the determination of organically combined sulfuric anhydride[8]. It consists essentially of extracting the original sample with a solvent over concentrated salt solution, purifying the extract, and weighing the residue. The fatty matter, which is considered to be practically unchanged in composition and weight from that in the original sample and consequently may be designated as the true fatty matter, is obtained from the residue by subtracting the organically combined sulfate group. The organically combined sulfate group in the residue is readily obtained by the usual methods.

The active ingredients in sulfonated oil may be indicated by the formula $ROSO_3Na$ where R represents the fatty radical. The question arises in calculating the fatty matter whether to subtract SO_3Na^- or SO_4Na^- from the residue. The true fatty matter in the original sample may be considered as RO, particularly in the case of castor oil where the oxygen forms part of the original hydroxyl group. In that event, SO_3Na^- should be used. On the other hand, the use of the factor SO_4Na^- is equivalent to representing the fatty matter as R—i.e., as ricinoricinoleic acid or similar polymers—and the fatty matter so determined more nearly corresponds to the decomposed fat, which contains a greater amount of polymerized compounds than the original sample.

Results by the Total Active Ingredients Method

A sample of sulfonated castor oil was analyzed by this method for true fatty matter by three different men and the results are given in Table III. All acidities in this paper were calculated on the original sample and then converted to milligrams of potassium hydroxide per gram of castor oil fatty acids, as determined by the Wizoeff method. The acidity of the original sample represents the free fatty acids plus the fatty acids as soap. In calculating the true fatty matter, the factor SO_3Na^- was used.

TABLE III

True Fatty Matter and Acidities by Total Active Ingredients Method

Analyst	Total Active Ingredients %	True Fatty Matter %	Acidities, mg. KOH per Gram Fatty Acids In True Fatty Matter	In Original Sample
1	71.32	....	....	....
2	71.43	....	....	....
3	71.29	....	....	....
Av.	71.35	64.65	117.6	119.2

The small drop from the original acidity indicates that the total active ingredients had probably undergone very little change during extraction and heating. Table IV shows that the total is within 0.2 per cent of 100 per cent.

TABLE IV

Complete Analysis of Sample of Sulfonated Castor Oil

	%
Moisture	25.1
Inorganic salts	0.9
(Na-H) as soap	1.2
Free glycerine	1.2
Total active ingredients	71.4
Total	99.8
Organically combined SO_3	5.21

For further determination of the purity of the total active ingredients, the following additional tests were applied:

Fatty Matter. The fatty matter in the total active ingredients was determined by the dilute-acid decomposition method and was found to check with the fatty matter in the original sample obtained by the same decomposition method. This was confirmed by the fact that the wash waters and salts upon the filter paper when decomposed by boiling with acid showed only a trace of fat.

Combined Sulfuric Anhydride. The combined SO_3 in the total active ingredients determined by the A. A. T. C. C.-acid titration method[8] agreed with the value obtained on the original sample, indicating no decomposition of the sulfonated oil.

Foreign Salts. The organically combined SO_3, determined by the A. A. T. C. C.-gravimetric method[8], checked with the combined SO_3 known to be present. Evidently the presence of foreign salt would have been indicated by a high result. The absence of foreign salt was also shown from the fact that upon chilling the residue after almost complete evaporation of the solvent before the addition of alkali, no separation occurred.

Solvent. The absence of solvent was proved by the fact that additional heating for over 4 hours at 110° C. had practically no effect on the constant weight.

Results by Different Methods

The same sample was analyzed also by the dilute-acid decomposition, Wizoeff, and strong-acid decomposition methods and the results, which are the averages of a number of tests, are listed in Table V.

TABLE V

Comparisons of Fatty Matter and Acidities Determined by Different Methods

Method	Fatty Matter %	Diff. %	Fatty Matter Based on T.A.I Method %	Diff. %	Acidities, mg. KOH Per Gram Fatty Acids
Original sample	....	...	....	..	119.2
Total active ingredients (T.A.I.)	64.65	...	100.0	..	117.5
Dilute-acid	63.68	0.97	98.5	1.5	133.0
Wizoeff	63.18	1.47	97.7	2.3	184.2
Strong-acid	62.63	2.02	96.7	3.3	96.1

It will be noticed that the total active ingredients method gave the highest results for total fatty matter and the strong-acid decomposition method the lowest—namely, 64.65 per cent compared with 62.63 per cent, or a difference of 3.3 per cent on the fat basis. The drop in acidity from 119.2 to 96.1 mg. in the strong-acid decomposition method indicates that the low result in fatty matter is partly due to polymerization whereby water is eliminated. Undoubtedly there was also some loss of glycerine. Upon comparing the dilute-acid decomposition method with the strong-acid decomposition method, the fatty matter was 63.68 and 62.63 per cent, respectively, and the acidity 133.0 and 96.1 mg., respectively. It is thus evident that the results by decomposition methods may vary with the conditions of decomposition. In the dilute-acid decomposition method, there was evidently a preponderance of hydrolysis of the glycerides over polymerization of the fatty acids, since the acidity is above the original sample; the reverse was true in the strong-acid decomposition method.

The fatty matter by the Wizoeff method lies between the two acid decomposition methods. The Wizoeff method yields a definite chemical compound—the pure fatty acids—and with care concordant results are readily obtained. The main objections to this method is that the results may differ. as already stated, from the true amount of fatty matter present in the original oil, depending upon the relative amounts of glycerides and polymers in the original sample. In the case of this sample, the fatty matter by this method was 2.3 per cent below the true fat as determined by the total active ingredients method. The difference may be even greater for a sample both high in glycerides and low in polymers, as for example, in a low sulfonated olive oil.

It will be observed that the highest result was obtained by the total active ingredients method. It is obvious that the method yielding the highest result must necessarily be the nearest to the true content of fatty matter unless splitting of the polymers occurs, which is considered not probable in acid solutions. Hence the fatty matter by

the total active ingredients method is considered the most accurate of the methods tested. Similarly, the order of diminishing accuracy of the other methods is considered to be dilute-acid decomposition, Wizoeff, and strong-acid decomposition.

It was also noticed that higher results were obtained by the original Herbig method, in which boiling with strong hydrochloric acid is continued for only 15 minutes undoubtedly because of less polymerization and hydrolysis. However, the turbidity of the contents after the short heating makes this procedure uncertain. The total active ingredients method was used to determine the fatty matter in samples of sulfonated olive oil, sulfonated tallow, highly sulfonated castor oil, true sulfonated fatty oil and sulfonated mineral oil, and the results were found to be as satisfactory as the analysis of the sample of sulfonated castor oil reported in this paper.

If $NaSO_4^-$ is subtracted from the total active ingredients instead of $NaSO_3^-$, the result for the sample analyzed checks closely with the dilute-acid decomposition method—namely, 63.60 per cent compared with 63.68 per cent, respectively. Hence as a matter of practical expediency the committee recommends the use of the factor $NaSO_4^-$ in comparing the fatty matter by the total active ingredients and the dilute-acid decomposition methods. The procedure by the former method is given at the end of this report.

TABLE VI

Total Active Ingredients in a Sample of Sulfonated Castor Oil

	Total Active Ingredients			
Analyst	*I* %	*II* %	*Av.* %	*Deviation from Mean* %
Ford	71.17	71.30	71.24	—0.07
Fowler	70.88	70.83	70.86	+0.31
Gulick-Segessemann	70.3	71.6	70.9 *	+0.27
Hart-Birnbaum	71.29	71.43	71.35*	—0.18
Pingree	70.92	71.96	71.44*	—0.27
Silverman-Winograd	70.7	71.9	71.45*	—0.28
Williams-Jarek	70.92	71.01	70.97	+0.20
Mean			71.17	
Highest			71.45	
Lowest			70.86	
Greatest difference			0.59	
Average deviation			0.23	

*More than 2 determinations.

COMMITTEE RESULTS

The committee analyzed a sample of sulfonated castor oil for total active ingredients in accordance with the proposed procedure and the results are given in Table VI. It will be noticed that the method yields concordant results, the greatest difference being 0.59 per cent and the average variation only 0.23 per cent. The total fatty matter was calculated from the total active ingredients by subtracting the combined SO_4Na^-, which for this sample was equal to 7.75 per cent, since the average results for the combined SO_3 previously found by the committee was 5.21 per cent. These values and the results by the proposed acid-decomposition method are given in Table VII. It will be noticed that the fatty matter by the two methods is within 0.5 per cent and that the greatest difference and the average variation by both methods, respectively, were practically alike.

TABLE VII

Total Fatty Matter in a Sample of Sulfonated Castor Oil by the Dilute-Acid Decomposition and the Total Active Ingredients Methods

	Total Fatty Matter — *Dilute-Acid Decomposition Method*			
Analyst	*I* %	*II* %	*Av.* %	*Deviation from Mean* %
Ford	63.71	63.60	63.66	+0.25
Fowler	64.18	64.16	64.17	—0.26
Gulick-Segessemann	63.7	63.9	63.8	+0.11
Hart-Birnbaum	63.59	63.77	63.68*	+0.23
Pingree	63.49	64.19	63.87*	+0.04
Silverman-Winograd	64.17	64.48	64.32*	—0.41
Williams-Jarek	63.86	63.92	63.89	+0.02
Mean			63.91	
Highest			64.32	
Lowest			63.66	
Greatest difference			0.66	
Average deviation			0.19	

*More than 2 determinations.

	Total Fatty Matter — *Total Active Ingredients Method*			
Analyst	*I* %	*II* %	*Av.* %	*Deviation from Mean* %
Ford	63.42	63.55	63.49	—0.07
Fowler	63.13	63.08	63.11	+0.31
Gulick-Segessemann	62.55	63.85	63.15*	+0.27
Hart-Birnbaum	63.54	63.68	63.60*	—0.18
Pingree	63.17	64.21	63.69*	—0.27
Silverman-Winograd	62.95	64.15	63.70*	—0.28
Williams-Jarek	63.17	63.26	63.22	+0.20
Mean			63.42	
Highest			63.70	
Lowest			63.11	
Greatest difference			0.59	
Average deviation			0.22	

*More than 2 determinations.

COMMENTS AND SUGGESTIONS

The following comments and suggestions were received from the members of the committee:

Arthur L. Fowler: I find the modified Herbig method (dilute-acid decomposition) to be entirely satisfactory; seemingly the right conditions are obtained only by refluxing with comparatively weak acid. I have run a number of samples using the total active ingredients method and found it perfectly satisfactory. I would recommend the total active ingredients method as the official method and the dilute-acid decomposition method as the alternative method.

Gulick-Segessemann: We have found by experience that results obtained by the original Herbig method are not comparable due to uneven and uncontrollable hydrolysis of the glycerides. In drying the total active ingredients we obtained constant weight by heating in an electric oven at 104° C.

Hart-Birnbaum: It was noticed that the fat determined by the acid decomposition method was very much darker if decomposed in an open beaker than if heated in a flask under a reflux condenser. The tendency of the total active ingredients to decompose upon drying (indicated by reddening of the methyl orange retained in the residue) was overcome by stabilizing with 2 cc. of N/2 alcoholic caustic potash. With that amount of alkali heating for eight hours at 110° C. caused no discoloration or darkening of the residue, although a decrease in the soap content had occurred; the effect on the result, however, is considered negligible. The total active ingredients, however, will not stand heating by the A.O.C.S. hot-plate method for moisture, as the oil discolors and chars. As the residue retains traces of moisture or alcohol tenaciously, rather prolonged drying is required to reach constant weight. It is also advisable to evaporate the alcohol as much as possible before adding the alkali otherwise the residue may jell and slow up volatilization of the solvents. If greater accuracy by the total active ingredients is desired, the combined salt solutions and the filter paper may be boiled with mineral acid and the separated fatty matter, if any, extracted and weighed.

R. A. Pingree: I frequently use the volumetric method for total fatty acids in soap and when carefully carried out, it serves to give relative results which at best are only roughly approximate. The A.L.C.A. method is subject to too many errors. I am also in accord with Mr. Segessemann's objections to the original Herbig method. In determining the fatty matter by the fatty acids method, there is a question in my mind as to whether any loss due to the condensation of the fatty acids would introduce an appreciable error into the results.

To determine the loss in weight of the extracted fat by the acid-decomposition method on prolonged heating, the samples were kept in the oven for a total of 29 hours and weighed at intervals. On plotting the time against loss in weight it was found that the loss was uniform during the entire period and, at the temperature of our oven (110° C.), amounted to approximately 3.2 mg. per hour. In two cases. the residues were dried according to the proposed A.O.C.S. method for moisture in oils—namely, as follows: Instead of prolonged heating in the oven, the fatty matter, contained in a 150-cc. tared beaker with short thermometer, was placed on a hot plate (after driving off the ether in a water bath), heated to 120° C., and maintained at this temperature (± 3° C.) for five minutes, stirring continuously with the thermometer. It was then placed in the oven at 110° C. for 15 minutes, allowed to cool in a desiccator, and then weighed. This operation was repeated several times and it was found that very consistent results could be obtained by this method, as the loss in weight after each successive heating and cooling amounted to only 1 mg.

In filtering the ether-oil solution, I find that a sintered glass filter is much superior to filter paper in that there is less creepage of the oil up the sides of the filter, and it requires much less ether to wash through the last traces of oil.

I believe that the T.A.I. method can be further simplified and made more fool-proof by eliminating the necessity of the final treatment of the ether-oil layer with a saturated solution of Na_2SO_4 and subsequent cooling in ice water. I believe that the same objective is obtained by shaking the ether-oil layer finally with anhydrous Na_2SO_4. This absorbs the last traces of moisture from the oil and allows it to be filtered directly into the tared beaker. In view of all the evidence obtained on these procedures, I am of the opinion that the total active ingredients method gives a more truly representative figure than the acid-decomposition methods.

Silverman-Winograd: There is no doubt that results by the original Herbig method may vary greatly, depending upon changes that take place during the splitting off of the combined SO_3 and also upon the degree of hydrolysis and polymerization. In the new method, we do not believe it should be referred to as "total active ingredients." The total purified sulfonated oil should be extracted, the combined SO_4Na^- determined by ashing the residue, and the difference between the two shall be reported as "total fatty matter." With the T.A.I. method, which determines the fatty matter as it exists in the original sample, it is possible for the first time to check an analysis of a sulfonated oil close to 100 per cent by adding the water, inorganic salts, combined alkali, and combined SO_4Na^-, tests for which are included in the A. A. T. C. C. methods of analysis. A large charge of the committee Sample C was extracted as in the active ingredients method and the glycerine extracted from the water layer. A residue representing 1.2 per cent of the

sample was obtained, which had a syrupy consistency, sweet taste and had the general characteristics of glycerine. Our analysis now totals as shown below:

	%
Total active ingredients	71.45
Inorganic salts	0.8
Alkali as soap as (Na-H)	1.2
Water	25.3
Glycerine	1.2
Total	99.95

No analytical difficulty was experienced with either method. The losses obtained upon drying the T.A.I. were:

	Loss, Grams
1 hour on steam bath and 0.5 hour in oven at 105° C.	....
Additional 0.5 hour in oven at 105° C.	0.050
Additional 20 minutes in oven at 105° C.	0.002

From the above it would seem that one hour on the steam bath followed by 1 to 1.5 hours in the oven at 105° C. would be sufficient to obtain constant weight.

Williams-Jarek: During the course of the work, the only difficulty encountered was the tendency of the fats, upon the evaporation of the ether, to creep over the walls of the beaker. This being particularly so in using a 50-cc. beaker in the total active ingredients method. To overcome this a flat-bottom extraction flask was used, which gave no trouble at all. With the amount of alkali added to the T.A.I., as specified in the proposed procedure, constant weight in drying was obtained in 2.5 hours without any decomposition setting in.

SUMMARY AND RECOMMENDATIONS

It was shown that the fatty acids or Wizoeff method for total fatty matter in sulfonated oils and the acid-decomposition or Herbig method yield low results. Furthermore, results by acid-decomposition methods vary with the method of decomposition—stronger acid and continued boiling yielding the lower values, probably due to greater polymerization. A new method for fatty matter, proposed by the committee and based on total active ingredients (T.A.I.), was found to yield accurate and concordant results. The weight of the fatty matter by the T.A.I. method probably corresponds to the true fatty matter as it exists in the original sample. The fatty matter determined by this method was in good agreement with the dilute-acid decomposition method—namely, 63.42 per cent compared with 63.91 per cent, respectively. Besides being based on a new principle, the proposed method is also applicable to true sulfonated fatty and mineral oils, in which cases the other methods, which depend upon acid-decomposition to render the fatty matter insoluble, cannot be used. Finally, it replaces with one determination the several tests required for grading sulfonated oils by the total active ingredients method as specified in Commercial Standard CS 43-32.

In view of these findings the committee recommends the total active ingredients method as a standard method for the determination of fatty matter in sulfonated oils and the dilute-acid decomposition method as an alternative. The procedures for both methods are given below:

PROPOSED STANDARD METHOD OF TEST FOR TOTAL FATTY MATTER IN SULFONATED AND SULFATED OILS

AATCC Total Active Ingredients Method

Scope:

1. This method of test determines the total fatty matter in a sample of sulfonated or sulfated oil as it exists in the original sample by extracting the undecomposed total active ingredients over a concentrated salt solution, from which the neutralized combined sulfuric anhydride is deducted.

Reagents

Ether:

2. Ethyl ether U.S.P. shall be used as the solvent.

Sodium Chloride:

3. The sodium chloride shall be C.P. and used in a concentration of 25 per cent.

Sodium Sulfate:

4. The sodium sulfate shall be C.P. and used either as anhydrous or in a concentration of 25 per cent.

Methyl Orange:

5. A 0.1 per cent water solution of methyl orange shall be used as the indicator.

Alcoholic Caustic Potash:

6. A standardized N/2 alcoholic caustic potash shall be used.

Procedure

General:

7. The procedure shall depend upon whether or not the sample upon shaking with ether and concentrated salt solution forms two or three layers.

Two Layers (Ordinary Sulfonated Oils):

8. Enough of the sample to yield about 5 grams of anhydrous fat shall be weighed into a 250-cc. pear-shaped separatory funnel containing 50 ml. of sodium chloride solution, some solid salt, 5 drops of methyl orange indicator and 50 ml. of ether. The mixture shall be shaken and neutralized with approximately N sulfuric acid until the lower layer is distinctly pink (about 0.2 ml. excess). After settling for at least 5 minutes, the lower layer shall be drawn off into another separatory funnel, and the ether layer washed with 25-ml. portions of the salt solution until practically neutral to methyl orange—i.e., until one drop of N/2 caustic soda turns the wash water strongly alkaline. All separations shall be allowed to settle for

at least 5 minutes. The water layers shall then be combined, extracted with two 25-ml. portions of ether, and the last two ether extractions combined and washed with salt solution until free from acid as with the ether layer in the first funnel. All the ether extracts shall now be combined, any water that may settle carefully removed, and the ether layer dehydrated with sodium sulfate either in the form of its anhydrous salt or its concentrated solution. To dehydrate with the solution, the ether layer shall be shaken well with 10 ml. of sodium sulfate solution, warmed to 55° C., for about two minutes. The mixture shall be allowed to separate for about 10 minutes and, to prevent crystallization of the salt, the lower part of the funnel shall be immersed in a beaker of warm water or otherwise heated. The lower layer shall now be removed as completely as possible, the ether layer transferred to a dry 250-ml. Erlenmeyer flask, the funnel rinsed several times with ether, and the solvent evaporated to about 75 ml. The Erlenmeyer flask shall be immersed in ice water for 30 minutes, with frequent mixing, and finally filtered into a tared 150-ml. beaker. The flask and filter paper shall be washed with ether until free from fat (absence of oil stains on filter paper upon drying) and the filtrate added to the beaker.

To dehydrate with the dry salt, 5 grams of anhydrous sodium sulfate shall be added to the ether layer, shaken vigorously for 5 minutes, and filtered directly into a tared 150-ml. beaker, placed in a water bath. The flask and filter shall be washed with ether until free from fat (absence of oil stains on filter paper upon drying) and the filtrate added to the beaker. To avoid creeping of oil, the beaker shall at no time be more than one-third full during filtering and washing.

The filtrate in either case shall now be evaporated to a total volume of about 20 ml., exactly 2 ml. of alcohol caustic potash added, contents mixed by swirling, and finally evaporated until practically free of ether. The residue shall be dried at 108° to 112° C. for 1.5 hours, cooled in a desiccator, and weighed. The heating shall be repeated for 30-minute periods until constant weight is reached.

**Three Layers (Highly Sulfonated Oils)*:

9. The sample shall be extracted over salt solution and the two top layers washed as under "Two Layers." The lower water layer and the washes shall be drawn off into a second funnel and extracted twice with a mixture of 20 ml. of ether and 10 ml. of alcohol. The combined ether-alcohol layers shall then be washed with 25-ml. portions of salt solution until neutral to methyl orange. All the extracts shall now be combined and washed with sodium sulfate solution as in "Two Layers." If a middle layer forms, 5-ml. portions of alcohol shall be added until it disappears. The ether-alcohol layer shall be evaporated, chilled and filtered as in "Two Layers," except that the flask and filter paper shall be washed with a mixture of 2 parts of ether to 1 part of alcohol. The greater part of the solvent shall be evaporated on the water bath, 1 ml. of alcoholic caustic potash added, and again heated on the water bath until practically free of solvent. Evaporation of the solvent may be hastened by stirring with a glass rod, which shall be tared with the beaker. Five ml. more of alcoholic caustic potash shall now be added and the residue heated to constant weight as in "Two Layers."

*This variation, which applies only to a limited number of highly sulfonated oils on the market, has not been tested by the committee but was thoroughly investigated in the chairman's laboratory.

Calculations

Correction for Alkali Added:

10. The correction for the alkali added in grams shall be the ml. of the alcoholic caustic potash, multiplied by (K-H), multiplied by the normality of the potash, and divided by 1000, or

Correction for alkali added,

$$\text{gms.} = \frac{(\text{ml. of KOH}) \times (\text{K-H}) \times \text{normality of KOH.}}{1000}$$

Correction for Neturalized Combined Sulfuric Anhydride ($NaSO_4^-$):

11. The correction for neutralized combined sulfuric anhydride ($NaSO_4^-$) in per cent shall be the ratio of $\frac{NaSO_4^-}{SO_3}$ multiplied by the per cent organically combined SO_3 in the original sample, or

Correction of neutralized combined sulfuric anhydride ($NaSO_4^-$), per cent $= \frac{NaSO_4^-}{SO_3} \times$ per cent combined $SO_3 = 1.2875 \times$ per cent combined SO_3.

Total Active Ingredients:

12. The weight of the residue minus the correction for the alkali added, divided by the weight of the sample, multiplied by 100 shall be the percentage of total active ingredients, or

$$\text{Per cent total active ingredients} = 100 \frac{(\text{weight residue minus alkali-correction})}{\text{weight sample}}.$$

Total Fatty Matter:

13. The percentage of the total active ingredients minus the correction for neutralized combined sulfuric anhydride ($NaSO_4^-$) shall be the percentage of total fatty matter, or

Per cent total fatty matter = Per cent total active ingredients minus per cent correction for neutralized combined sulfuric anhydride ($NaSO_4^-$)

and shall be reported as ". . . per cent total fatty matter by weight, A. A. T. C. C. Total Active Ingredients Method."

Accuracy

Accuracy:

14. Tests by the committee indicate that the average variation of a single analysis of a commercial 75 per cent sulfonated oil may be expected to be about 0.25 per cent.

PROPOSED ALTERNATIVE METHOD OF TEST FOR TOTAL FATTY MATTER IN SULFONATED (SULFATED) OILS

A.A.T.C.C. Acid-Decomposition Method

Scope:

1. This method of test determines the total fatty matter in a sample of sulfonated (sulfated) oil by decomposition with dilute mineral acids and extraction of the decomposed fat. This method is not applicable to samples that are not completely decomposed upon boiling with mineral acids.

Reagents

Acid:

2. The acid shall be approximately N sulfuric acid.

Ether:

3. Ethyl ether U.S.P. shall be used as the solvent.

Procedure

Procedure:

4. Enough of the sample to yield about 5 grams of anhydrous fat shall be weighed into a 300-ml. Erlenmeyer flask, provided with an air condenser and containing perforated beads. Enough sulfuric acid shall be added to decompose any soap that may be present and to contain an excess of 25 to 30 ml., and the contents boiled with occasional shaking for 1.5 hours, or until both layers are clear. After cooling, the contents shall be transferred into a 250-ml. separatory funnel and shaken with 50 ml. of ether. The water layer shall be drawn off into another separatory funnel and extracted twice with 25-ml. portions of ether. The combined ether layers shall be washed with 15-ml. portions of water until the wash water is neutral to methyl orange. The ether layer shall now be transferred to a tared 150-ml. beaker, evaporated on the water-bath until practically free of solvent, dried in a hot-air oven at 105 ° to 110° C. for 30 minutes, and finally cooled in a desiccator and weighed. The heating shall be repeated for 30-minute periods until constant weight is reached.

Calculation

Total Fatty Matter:

5. The weight of the residue, divided by the weight of the sample, and multiplied by 100 shall be the percentage of total fatty matter, or

$$\text{Per cent total fatty matter} = \frac{\text{Weight residue}}{\text{Weight sample}} \times 100$$

and shall be reported as ". . . per cent total fatty matter by weight, A. A. T. C. C. Acid Decomposition Method."

Accuracy

Accuracy:

6. Tests by the committee indicate that the average variation of a single analysis of a commercial 75 per cent sulfonated oil may be expected to be about 0.25 per cent.

ACKNOWLEDGMENT

The chairman is greatly indebted to Mr. I. Silverman of L. Sonneborn Sons, Inc., for the determination of free glycerine in the sample and to the members of the committee for their painstaking efforts with this assignment.

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ONE HUNDRED AND FIRST COUNCIL MEETING

April 12, 1935

THE Council held its 101st meeting at the Bureau of Standards in Washington, on Friday afternoon, April 12, 1935. Present were President Robert E. Rose, presiding; William R. Moorhouse, Treasurer; Louis A. Olney, Chairman of the Research Committee; William D. Appel, Hugh Christison, Henry F. Herrmann, and Donald H. Powers, Councilors at Large; Roland E. Derby, representing Northern New England and Eugene C. Knaeble, Philadelphia; and Harold C. Chapin, Secretary.

The Secretary's report of the last meeting was approved as published. The Treasurer's report of April 11, and the Secretary's balance sheet of April 11, were accepted. At suggestion of the Treasurer it was moved and voted that hereafter interim reports of the Treasurer should not be published, because receipts other than from the Textile Foundation were evident from the Secretary's balance sheets, and because expenditures throughout the fiscal year from funds collected at its beginning, could be shown in proper relation to one another only in the final report.

A new and in some respects lower schedule of prices for back numbers of the American Dyestuff Reporter was approved. They range, postpaid, from fifty cents for a single copy to $5.25 for a year, to non-members; and from thirty-five cents for a single copy to $4.50 for year, to members of the Association.

Mr. Herrmann suggested making the Association more attractive to textile printers. He was directed to make further investigation and report.

A program for the Research Associates in the year beginning with April, 1935, was presented by the Research Committee and approved. It was voted that if this should receive the complete financial support requested of the Textile Foundation, there should be appropriated from the Association's own Research Fund, $1,200 for Mercerizing Research.

Mr. Christison suggested that Council and Research Committee meetings might be held at a later hour on Fridays, or on Saturdays, at less expense to members for transportation, and possibly at greater convenience in other ways. The Secretary was directed to investigate and report to the President, so that the next meeting, planned to be in Boston on June 7 or 8, might be called in accordance with the desires of members.

Each of the following was elected to the class of membership specified, as of thirty days from publication of application, provided that no objection be received before that date by the Secretary.

Active

Georg Altenbaumer, Harry Bennett, Wm. H. Bertholet, Jr., James E. Bolderson, William Brandenburger, William L. Braun, C. A. Cates, J. Reese Daniel, James V. DeMick, E. H. Dobbins, Joseph Frank, Eugene E. Gansel, Ellis Greene, Albert E. Herrmann, Jr., Elmer Hulton, Clyde E. A. Keene, William M. Kirk, Loring P. Litchfield, William B. Luce, H. Grady Miller, Harold Mudd, John B. Neely, Ralph H. Pfeiffer, Frank R. Piper, Henry C. Schuffels, J. E. Smenner, Harry E. Smith, Samuel C. Smith, Alfred M. Townson, Charles P. Walker, Herman Andrew Walker, W. A. Wright, L. D. Wyrick, Edvard Young, Eric Martin Ziegler.

SECRETARY'S BALANCE SHEET

April 11, 1935

	Applications	Dues, Regular & Re-instate.	Dues, Sustaining & Contributions	Dues Corporate	Miscellaneous	Bank Charges	Totals Net
Received by Secretary, Nov. 1, 1934 to Feb. 27, 1935.....	$640.00	$5,130.00	$225.00	$1,575.00	$893.77	—1.72	$8,462.05
Received by Secretary, Feb. 27 to April 11, 1935.....	257.50	562.25		100.00	55.40*	— .51	974.64
Total receipts for fiscal year to April 11, 1935.....	897.50	5,692.25	225.00	1,675.00	949.17	—2.23	9,436.69
Transmitted to Treasurer	852.50	5,355.00	225.00	1,675.00	925.94	—1.97	9,031.47
Leaving to order of Secretary in Appleton Bank, Lowell...	45.00	337.25			23.23	— .26	405.22

***Miscellaneous Items**

Year Books	$27.69
Analytical Methods	20.01
Dyestuff Reporters	4.70
Pins	3.00
Total	$55.40

Junior

Melville J. Blackwood, Thomas Sydney Blackwood, James P. Conaway, J. L. DeBatin, Joseph Miles Edwards, Jr., W. C. Magill, A. A. Mailloux, John Wesley Miles, J. A. Mott, Jr., B. B. Newlin, J. W. Stallings, James Tinkler.

Associate

Alan W. Burke, W. Warren Doernbach, Arthur Finley, Benjamin T. Leaver, George Schultz McCarty, Elmer W. Schmalenberger.

Student

H. S. Anthony, J. M. Fletcher, Lee Gale Johnston.

Francis T. Seery and Mike Stough were transferred from other classifications to Active membership.

Respectfully submitted,

HAROLD C. CHAPIN,
Secretary.

FEBRUARY MEETING, NEW YORK SECTION

THE February meeting of the New York Section was held on Friday evening, March 1st, 1935 at the Alexander Hamilton Hotel in Paterson, N. J.

Harry E. Smith of the Clarion Chemical Corp. presented a paper on the subject of "Pine Oil and Pine Oil Penetrants as Used in the Textile Industry."

H. C. Chapin, national secretary of the Association, was present and addressed the meeting.

Respectfully submitted,

P. J. KENNEDY,
Secretary.

MARCH MEETING, NEW YORK SECTION

THE March meeting of the New York Section was held at the Chemists' Club, New York City, on Friday evening, March 29, 1935.

M. C. Teague, of the Naugatuck Chemical Co., a division of the U. S. Rubber Co., presented a paper on the use of latex in the textile field.

A motion picture entitled the "Romance of Rubber" was shown and N. H. Brewster, also of the Naugatuck Chemical Co., gave a talk while the picture was being shown.

Respectfully submitted,

P. J. KENNEDY,
Secretary.

STANDARD SILK SAMPLES

There have been prepared, under the direction of the Sub-Committee on Fastness of Dyed Silk, standard dyed silk fabrics to represent four classes of fastness to washing—namely, Class I, Class II, Class III and Class IV. These standards have been carefully dyed with the dyestuffs and according to the dyeing methods recommended in the 1931 Year Book.

The A.A.T.C.C. is prepared to furnish sets of these washing standards for a nominal charge which will cover the cost of preparation. It will be possible to accurately grade the fastness by comparing it with the standards after subjection to the standard washing tests approved by this Association. All inquiries concerning these Silk Washing Standards may be addressed to the Chairman of the Research Committee.

Louis A. Olney,
Lowell Textile Institute,
Lowell, Massachusetts

COMMUNICATION CONCERNING 1934 YEAR BOOK

To All Members of the Association:—

THE 1934 Year Book is nearing the point where it will be possible to put it to press. The text matter is very largely in hand, but the advertising ordered has not as yet been sufficient to pay the cost of production. This letter is therefore addressed to all members in the hope that they will cooperate in an effort to bring the revenue up to a point which will make it possible to at least pay the cost of production, if not to provide a profit for the use of the Research Committee, as was the case last year.

There are undoubtedly many members who are connected with firms who might logically use advertising space in this volume or who are acquainted with such firms. Appended below is a list of all the concerns who have engaged advertising space at this writing. If members will scan this list they will undoubtedly find many firms with whom they have influence who are logical advertisers but who are not as yet represented. In such cases, we will appreciate the members doing two things—

1. Notify the undersigned of the name and address of the prospective advertiser.
2. Drop a line to the advertiser or otherwise communicate with him, telling him of the advantages of advertising in the Year Book.

When it is taken into consideration that copies of the Year Book go to each member of the Association, which now includes nearly 1,500 individuals, and that these men control the purchase of chemical supplies and equipment for a very large percentage of the textile industry of the country, the advertising value of the publication is unquestionable. It should further be borne in mind that this volume, unlike the annual publications of many Associations, is in reality a technical reference book which is constantly used by members rather than merely a list of members.

The following advertisers have thus far contracted for space in the 1934 Edition.

(Signed) L. A. OLNEY,
Chairman, Year Book Committee,
Lowell, Mass.

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American Aniline Products, Inc.
American Aniline & Extract Co.
American Cyanamid & Chemical Corp.
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American Potash & Chemical Corp.
Amid Duron Co.
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INTERPRETATION OF RESEARCH—II
What Shall be Interpreted?

(Part I appeared on page 257 in our last issue)

IN Part I we recognized that there are various sorts of material that need to be interpreted in order to be applied in the textile industry. It seems that one of the real needs is the interpretation of some very "scientific" data into terms of textiles. What is this, however, but the seeking of textile applications for scientific knowledge?

The scientific research man who is approaching textiles may need a little interpretation the other way around but, speaking generally, is not one of our greatest needs the correlation of the advances of science with our industry so that the men in it of limited education or opportunity for study can understand the value of the science and perhaps see that it is applied?

Another aspect to the interpretation of research is that there is already at hand a vast amount of research information in published form and that it should be made available and should be applied more generally throughout the industry. Undoubtedly sufficient study and the adoption of much that is already known would be of great value to many mills. It is a proper task of research to collect this material, check its correctness, explain or interpret it where necessary and put it at the disposal of the textile industry. Undoubtedly a vast amount of this research would have to be done over, some of it repeatedly so that the facts would be thoroughly established.

Then, no matter in what wonderful and useful form this material might be presented, it would be completely ignored by a great share of the industry. No amount of interpretation would get it as widely used as it should be and the natural argument would be raised that we don't use what we have so why bother with more science?

The answer is that science is *usually* far ahead of practice. It seems inevitable that even though both advance there will be a great unclosed gap between them. We want to close the gap but we want to keep on going ahead at the same time. Hence let us have all the research that is possible but try to keep up the interpretation at a little greater rate all the time and gradually catch up.

The study of research data and results in order to understand or utilize everything of value is one of those things which, from its very nature, can never be completed. If the work is at all complicated or extensive it cannot be interpreted once and for all. Usually one can go over it again and again and always find something new.

The studying of old data is like the performing of experiments. Almost invariably you can see how to do better or to get more out of the experiment if you were to do it again. Published work is similarly incomplete, but as knowledge increases, sometimes you can see more and more in old data and thus make it of more use.

The sad thing is that interpreting does not necessarily mean practicing.

Also, there are times when it seems better to forget all that has been published in the past and to start over again with the present day knowledge of science, and to try to work out the interpretation and application to the textile industry with no preconceived ideas or prejudices.

There is one belief that seems to be fairly widely held, that research can be divided up in some systematic manner and completed a unit at a time. We do have to divide it up and perform a section at a time but when once "done" it is not necessarily done for good. Some new research tool may enable us to do it over again with much better results, then there may be a new interpretation and new applications.

Of course this all leads up to the value of science and research in the mill. One of its great objectives is to promote the understanding of things as they already are.

There is always that fear in the mill represented by the words, "Don't change anything". It is well grounded on experience but should we not know enough about our work to know at least why we should not make changes? We all know of processes with details which are vital to success, but in other cases probably a good many of our fussy details are of no value at all but we don't *know* enough to change or drop them.

Experience has shown that as one gets to know a process he does not fear to try little changes in it because he can see what a small change in one of the variables is likely to do and play safe while feeling it out. Frequently some of the details and precautions will be found to be useless and an unnecessary expense. Cautious study of the variables and the correct interpretation of their effects may finally lead to definite improvements in a product.

Study, understanding and interpretation are so similar and so closely related that it is difficult to make any distinction between them.

COTTON SHIRTING FABRIC

(Continued from page 268)

In order to obtain the utmost penetration of the fabric by the caustic soda solution yet another expedient can be adopted. There is some doubt as to the best temperature for the alkali in order to obtain the utmost luster. On one hand it is stated that the caustic soda solution should be as near freezing point as possible and on the other there are records that excellent luster can be secured by the use of the alkali solution at temperatures up to 60°C. Whatever may be the truth in these records, and it is possible that both high and low temperatures can be made to give good luster results if the other conditions of mercerization are suitably modified, there is no doubt but that increased penetration is obtained with the high temperature mercerizing liquor. Also, in the writer's experience, under certain conditions, the mercerizing liquor is best maintaned at about 15° to 20°C. But having two mangles available it is possible to use both high and low temperature mercerizing liquors.

It is recommended to have in the first impregnating mangle caustic soda at about 30° to 40°C. While the fabric is led over the upper cylinders it cools considerably. Then if cold caustic soda solution is contained in the second mangle the fabric is completely cooled to room temperature. Thus the first mangle with its warm caustic soda effects good penetration and the second mangle with its cooled liquor ensures that the fabric is brought to a lower temperature at which most luster is produced.

The concentration of the mercerizing liquor is satisfactory when the liquor is of 52°Tw.

It is, of course, most desirable to run the mercerizing machine at the highest possible speed in order to obtain a good output of mercerized fabric. By using two impregnating mangles higher speeds can be obtained than when only one mangle is used, for the caustic soda has time to act on the cotton fibers while the fabric is being led over the upper cylinders placed between the two mangles. Also, with two mangles good penetration is ensured although the time that the fabric is squeezed between the bowls of the mangle is shortened. Provided that the stenter frame is sufficiently long to allow satisfactory washing out of the alkali from the fabric it is possible to mercerize at 60 yards per minute. From practical experience it is considered extremely likely that this speed could be increased to 70 yards per minute without reducing the luster increase.

In this article details of the arrangements for washing out the alkali from the fabric will not be discussed since these do not materially affect the luster or handle of the fabric.

As previously indicated, the fabric is soured after leaving the stenter frame, it being found extremely difficult to remove the last traces of alkali without treatment with an acid. But a subtle point arises in connection with this acidification. It is found that the process affects the handle of the fabric, generally to make it somewhat harsher.

In considering this point it must be noted that cotton fibers saturated with caustic soda of mercerizing strength are brought into a highly swollen and plastic state. If the fibers be treated with a strongly acid solution neutralization of the alkali takes place and the fibers rapidly contract. This phenomenon can be likened to the immersion of a sheet of glue in water. The glue swells a great deal without dissolving. If the sheet of swollen glue be now dried it collapses until it becomes a hard sheet once more. In the mercerization process the cotton fibers swell until they acquire the soft swollen form of the sheet of glue and if the fabric is to have a super soft handle an endeavor should be made to wash out the alkali and dry the fabric with the least contraction and hardening of the swollen cellulose.

Acidification brings about a rapid collapse of the swollen cotton fibers. The experiment has been made of neutralizing the residual alkali in a slower manner. Instead of a dilute solution of hydrochloric or sulfuric acid there was used a solution of sodium bicarbonate in one instance, and boric acid in another. In the case of sodium bicarbonate it was found that the fabric had a softer handle than usual, and although it was not possible to show that the cotton fibers were in a more highly swollen state than when sulfuric acid was employed it was believed that such was the case and was the explanation of the softer handle.

With boric acid no marked difference of handle could be detected. Also the use of boric acid as a neutralizing agent is less convenient than sodium bicarbonate. It will be remembered that the washing liquors are collected and causticized with milk of lime to form caustic soda, and after concentration these liquors are again used for mercerization. Thus any sodium bicarbonate used is first converted into sodium carbonate in the washing thus

$$NaHCO_3 + NaOH = Na_2CO_3 + H_2O$$

and this, in the alkali recovery process is re-converted into caustic soda according to the following equation

$$Na_2CO_3 + Ca(OH)_2 = 2NaOH + CaCo_3$$

There is thus no loss of the sodium bicarbonate.

It is suggested that those interested particularly in softness of handle in mercerized cotton goods will find well worth study the conditions under which the alkali is removed from the treated fabric for it is believed that in this process the handle is affected much more than is commonly suspected.

After mercerization it is necessary to examine the colored stripes and to note how much they have changed. The usual effect is that the colors have deepened, but this general change is sometimes accompanied by slight variations in tone, particularly the blue colors being greener. In order to correct these changes the fabric should be given a short boil in dilute soap solution. This treatment also has the effect of brightening the colors.

After these treatments it is frequently found that the blue colors are yet too green. There is yet another corrective treatment which can make these redder. The fabric should be boiled for about ten minutes in a dilute solution containing soda ash and glucose. Instead of this a warm very dilute solution of sodium hydrosulfite can be employed but care must be taken that this does not affect the colors other than blue. On the whole, the alkaline glucose liquor is safer and is to be preferred. In this treatment the blue vat dye is slightly reduced and in this process it loses its greenish tone.

3. FINISHING

Having brought the colored stripes to their original shade it is now necessary to finish the fabric. If a fully guaranteed unshrinkable finish is demanded and one or other of the patented finishing machines for this purpose is available then of course this machine should be used. But if the finish is just required to be practically, without being absolutely unshrinkable it is possible to achieve this end with the ordinary pin stenter provided that certain expedients are adopted. These will now be discussed.

Firstly, the fabric should be brought into a fully shrunken state before it is stentered. Two methods are available. In the first, the fabric is placed quite loosely in a rotary machine and there boiled for about 15 minutes with a dilute soap solution. Under these conditions the fabric shrinks up much as it might do when afterwards made up into a shirt and laundered. Alternatively, the fabric can be treated for 20 minutes in a winch machine with a similar soap liquor. Although the fabric in this machine is repeatedly drawn out of the liquor and over the winch it is found as a matter of practical experience that nearly as much shrinkage is obtained as in the rotary machine. Following either of these treatments the fabric should be hydroextracted and opened out to full width preparatory to stentering.

In the stentering, the all-important feature is the manner in which the fabric is delivered on to the stenter pins and the type of pin plates used. By skilled use of the stenter it is possible to contract the fabric in length so that it is substantially unshrinkable in subsequent laundering. This applies mainly to shirting fabric of the usual thickness. It is found that the thicker and less flexible is the fabric the more difficult it is to contract it on the stenter.

Before considering a special type of pin plate which is favorable to fabric contraction and which has been devised by the writer, attention will be drawn to the commonly used method for securing contraction by guiding the fabric on to the pin plates on a curved surface. Referring to Fig. 2 there is shown a chain of pin plates A, B, C, D, E, F, etc., being drawn around the guiding wheel W at the end of the stenter where the fabric P enters. On each plate is shown one pin on which the fabric is impaled so that it may be carried forward through the hot air chamber H and so dried while being stretched to its required width. It is at once evident that if the fabric is impaled on the pins while passing around W then the length of fabric *a* between consecutive pins is greater than the distance *b* between the pins when they leave the wheel W and travel straight. Because of this, the fabric takes a crinkled form on the pin plates as it passes forward to the hot air chamber. During the drying these crinkles straighten out and the fabric thus becomes contracted and substantially unshrinkable. This principle is utilized in E.P. 346,782 in a somewhat special manner but the principle is well known to fabric finishers and is used in various modifications.

A somewhat ingenious modification makes use of the spaces between consecutive pin plates rather than the pin plates themselves. This is shown in Fig. 3. As the fabric P is fed on to the pin plates A, B, C, etc., while passing around the wheel W (Fig. 2) a brush presses the fabric downwards so that quite a pucker of fabric is formed be-

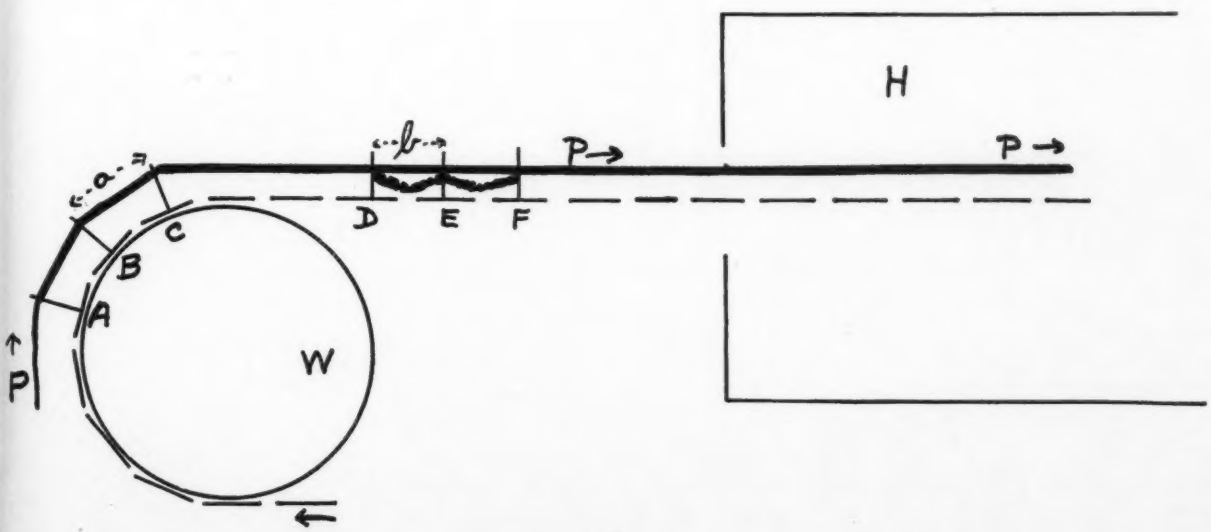


Fig. 2

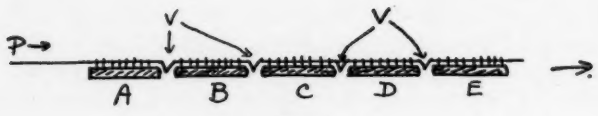
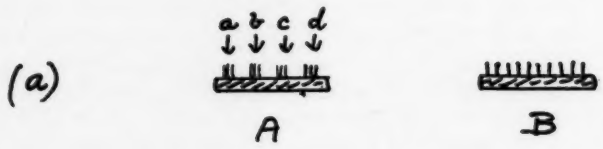


Fig. 3



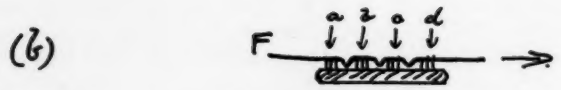


Fig. 4

tween each pair of plates as shown at V. In the hot air chamber these puckers straighten out as previously indicated and so produce length contraction.

Turning now to a more novel method the pin plate specially designed for the purpose is shown in Fig. 4*a*, an ordinary pin plate being shown at B by way of comparison. It will be noticed that the pins on plate A are arranged in groups *a, b, c,* etc., while those on plate B are evenly spaced. Moreover, the pins in the groups on plate A are spaced considerably more closely than those on plate B. In using a stenter provided with these special pin plates the fabric is fed in on the flat (that is, it is not necessary to feed the fabric on to the pins while these are passing around the wheel W as shown in Fig. 2) and is immediately pressed down quite firmly with a revolving brush. Owing to the resistance of the pins towards fabric being impaled upon it the fabric is well puckered in the spaces between the groups of pins *a, b, c,* etc., as shown in Fig. 4*b*. Quite a large amount of length contraction of the fabric can be obtained by using this special type of pin plate and by varying the amount of pressure on the impaling brush this contraction can be modified within fairly wide limits.

From the above, it is evident how much depends on the manner of conducting stentering when it is required to secure a finished fabric fully contracted in length so that it can be regarded as practically unshrinkable.

After stentering, the fabric usually requires a very light calendering in order to increase its softness but a high pressure on the fabric while passing between the bowls of the calender must definitely be avoided since such pressure lengthens the fabric and thereby makes it shrinkable in later washing.

Since the fabric must be soft and silky it is desirable to impregnate it with a softening agent immediately before stentering. This agent should preferably have a lecithin basis since waxy softening agents frequently cause the threads in the fabric to slip badly.

The above description of bleaching, mercerizing, and finishing cotton colored shirting fabric is based on practical experience and if the methods described are followed then there is obtained a highly lustrous fabric which has a very attractive appearance and a pleasing handle.

● GENERAL RELEASES

General Dyestuff Corp. announces release of circulars on the following products. Copies of the circulars are available upon request.

Naphtol A S Dyeings on Silk—a sample card showing a large range of shades suitable for this class of work. The dyeings are said to be fast to boiling, degumming and bleaching.

Benzo Fast Heliotrope 2 R L A—a direct dyestuff giving bright shades of Helio on rayon and cotton and somewhat redder shades on silk.

Stilbene Yellow G A Conc.—a direct color for dyeing cotton or rayon, which is said to stain wool or silk but very slightly from a neutral bath and to leave acetate clear.

Wool Fast F F G A—a new acid dyeing blue manufactured by General Aniline Works. It is said to be of very good fastness to fulling, washing, salt-water and perspiration and good fastness to light. It is said to be distinguished by its bright, greenish blue shade which does not turn red under artificial light. The shade is said to be only slightly dulled by chrome. It is stated that this dye exhausts well from a neutral bath and has good affinity for pure and tin weighted silk. It is further said to be very stable to Rongalite and therefore suitable for colored discharge work.

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WANTED: Expert cotton dyer thoroughly familiar with operation of package dye machines, preferably Obermaier. Good executive and thorough knowledge of application of dyestuffs. Please reply stating qualification fully, experience and past connections. Box No. 859. American Dyestuff Reporter, 440 Fourth Ave., New York, N. Y.